Sampling under Duress – What Few Want to Say and Even Fewer Want to Hear

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ABSTRACT

Key topics addressed in the present contribution: i) use and misuse of the Assay Exchange paradigm; ii) manipulating mine, ore and metal processing sampling for increased profitability by 'tuning' sampling and assaying; iii) sampling for refining and metal accounting; iv) risk mitigation 'games'. To paint the backdrop for the many ways to lose one's righteous way, I first present a detailed analysis of the assay exchange formalism, the 'scheme', which is involved in most trading and commercial contractual transactions. Based on a thorough Theory of Sampling (TOS) analysis, it is revealed how no less than two hidden, very often un-recognised sampling and sub-sampling stages 'before assaying' play a crucial role in determining the reliability of the final analytical determinations, demolishing the notion that total Measurement Uncertainty is a matter of analysis/assaying only. There are many tempting opportunities for devious behaviour and lack of due diligence along the 'lot-to-assaying-to settlement' pathway, ranging from ill-informed negligence to deliberate fraud. Presented here are some of the more gregarious examples of sampling under duress – and what can be done about it. Indeed, what should be done about this is simplicity itself, exclusively and honestly relying on the Theory of Sampling (TOS) only, with voluntary full transparency.

1. Introduction

At a recent conference three gentlemen of science, technology, industry and commerce accidentally found one another in agreement on a not so gentleman-like three-pronged theme: "Use and Misuse of the Assay Exchange paradigm" – "Manipulating mine and process metal accounting" - "Increasing profitability by 'tuning' the sampling and assaying for exchange". What do these topics have in common? On closer scrutiny, it turned out that all is not always right in the sampling realm!

Most individual experiences show that professionals and technical staff involved in sampling of ores, concentrates, refined metal products (f.ex., PGM), recyclates, spent catalysts and complex secondary mixtures all attempt to do the best job possible.

However, such best efforts are sometimes subverted by actions of the few that fully understand the mechanics of sampling and assaying and where to apply pressure to achieve 'more beneficial results' at particular stages in the logistics and processing chain from ore-to-refined and traded metal. This is unfortunately deliberate dishonest behaviour!

Below follows a sketchy trace of the origin and many manifestations of such misbehaviour, expounding their negative effects and impacts – but ending with showing the only, completely honest way forward.

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2. What caused this? – Part 1

The following conversation was conducted by the author in his erstwhile role as professor with an all-consuming interest in sampling. Knowing very well the necessary-and-sufficient principles behind representative sampling (having taught the discipline of the Theory of Sampling (TOS) for more than two decades), the question is: why are these principles not always followed where they demonstrably matter, for example where even very small deviations between true and estimated lot, batch, or consignment concentrations translate into highly significant economic gains or losses?

Think of industry sectors dealing with Gold, Silver, Platinum Group Metals (PGM), REE, critical minerals, large quantity commodities even? The following is a reflection by a very experienced agent at the front line where resource and commodity trading meets TIC companies (Testing, Inspection, Certification) offering services to this sector.

Upon direct questioning a relevant TIC-representative, the following statement resulted:

Evidence from the trenches

"Representatives from TIC companies feel that full application of TOS in the field can be challenging, at least when sampling commodities and minerals of high value. This relates to when stipulating sampling procedures to inspectors operating in remote locations around the world, on spot assignments with limited access to the whole consignment and with challenges in extracting a representative sample.
The major challenge is to convince clients, a lot of them traders, to spend the necessary efforts - read *money* - to allow to open packaging for full access or pass the material through an automatic sampler to take a larger sample mass. I guess this applies to any industry. When global traders put their negotiation skills to use with us, the humble TIC service providers, they win. I mean, they win because we will compromise on the representativity of the sampling and play by their rules. But in the end, we all lose as the results from the labs will be biased. And then we will spend time investigating and dealing with a claim. Of course, we add the necessary remarks and caveats in our reports and certificates, but it is all a game. The game they play is to save face with their buyers by pretending their product is of high quality, but that the opponent's 'sampling' and/or 'assaying' were done incorrectly. The other game they play is to use our knowledge of sampling to manipulate analytical results to their advantage. For example, finding out that natural segregation exists in high grade Cu concentrates, they try to manipulate the sampling technique into making a profit.

The more we educate such clients and share our knowledge, the more we are dragged into becoming puppets. In this context of dynamic and complicated mechanics of the global minerals trade, TOS is too often ignored, and the industrial practicians are many times ignorant of the work the professional and theoretical samplers are putting into creating a TOS framework that will benefit the whole industry.

Experience with practical application of TOS in the field is a thing that may, or may not (often), also apply to commercial settlement aspects in the business realm. We are often asked to give training to operational teams in different trading houses w.r.t. to different topics of concern, mainly to explain the differences in weighing and sampling between two ends: pit vs. furnace – or loading port vs. discharge port.

I believe partnering with independent TOS experts would greatly contribute to the necessary education of the relevant industries.

[The veracity of this statement is beyond reproach. The rapporteur, known to the author, shall remain incognito. It is not who said this, it is what is revealed that is important]

3. What caused this? – Part 2

At approximately the same time a fascinating book was recommended to the author: The Secret Club That Runs the World – Inside the Fraternity of Commodity Traders by investigative reporter and author Kate Kelly.

Who is Kate Kelly?

"I look for ways to shine a light on the inner workings of policy and governance. If there are significant amounts of capital or lever-pulling shaping political, legislative or regulatory outcomes, I'm interested."
https://www.nytimes.com/by/kate-kelly

A few snippets from publisher blurbs about this book will set the scene:

"Nestled deep in the towers of banking and finance are the commodities traders who spend their days gambling with oil, gold, and corn a.o. contracts. They're highly educated world travellers with a penchant for risk, and they're here to bet big on the future of the raw materials that make our economies hum. They're very wealthy, barely regulated, and can be a force for tremendous good--or for ill!"

"Commodity players are a shrewd and indomitable lot. And the contracts they trade are still so loosely regulated that the correct combination of money and skill creates irresistible opportunity. That's why Kate Kelly is only half joking when she calls them the 'secret club that runs the world'. When most people think of the drama of global finance, they think of stocks and bonds, venture capital, high-tech IPOs, and complex mortgage-backed securities. But commodities? Crude oil and soybeans? Copper and wheat? What could be more boring? That's exactly what the elite commodity traders want you to think. They don't seek the media spotlight. Their astonishing wealth was created in near-total obscurity, because they dwelled either in closely held private companies or deep within large banks and corporations, where commodity profits and losses weren't broken out. But if the individual participants in the great commodities boom of the 2000s went unnoticed, their impact did not. Over several years the size of the market exploded, and so did prices for raw materials—raising serious questions about whether the big traders were intentionally jacking up costs ... What was really driving all those price spikes? Kate Kelly takes us inside this secretive inner circle that controls so many things we all depend on. She gets closer than any previous reporter to understanding these whip-smart, aggressive, and often egomaniacal men (yes, they are nearly all men). They work hard, play hard, flaunt their wealth, and bet millions every day on a blend of facts, analysis, and pure gut instinct".

The present article is particularly interesting in these 'facts' and 'analytical/assay results', which are always considered fully reliable.

But are they?

4. Use and Misuse of the Assay Exchange paradigm

I admit that the above two information sources spurred more than a fleeting interest in these matters. But what really hit the head on the nail soon hereafter was being presented with a description of the well-known Assay Exchange 'mechanism'[2]. The Assay Exchange description was of such superficiality and showed a debilitating lack of the necessary scientific underpinnings w.r.t. the principles behind proper representative sampling, the Theory of Sampling (TOS). The present author therefore decided to delve into the depths of issues not before visited, namely: What is the origin and rationale behind the Assay Exchange paradigm and, how it is used today?

Two months' detective work then quickly resulted in an expository paper, aptly titled - also for the preset purpose: "The Elephant in the Room: Unsubstantiated Complacency Regarding the 'Assay Exchange' Paradigm – Sampling Uncertainties with Hidden Economic Consequences" [1,2].

4.1 Key message

"The assay exchange paradigm is an integral element in many contractual agreements stipulating how business transactions rely on comparison of two independent assay results for commercial and trading settlement purposes. It turns out that the current Assay Exchange vs. splitting gap paradigm incurs no less than two un-recognised sampling uncertainties, which leads to hidden adverse economic consequences for at least one – and sometimes for both – contractual parties. The magnitude of this unnecessary uncertainty is never estimated, which leaves management and business stakeholders without information about potential economic losses – a clear breach of due diligence.

2 This was parlayed by a much used 'expert' in another context, the specifics and details of which are of no relevance for the present exposé, but it revealed a seriously flawed understanding that called for more attention.

However, all that is needed to resolve this critical issue is stringent adherence to the Theory of Sampling (TOS) by mandatory contractual stipulations of only accepting representative sampling and sub-sampling principles, leading to a community call to action" [1,2].

4.2 Technical denouement

The elephant in the room is the tacit, unwarranted assumption that the contractual splitting gap is always centred on the true average lot concentration. Note for example in Figs. 2-5 below that an acceptable settlement assay is easily reached via the assay exchange scheme regardless of whichever analytical concentration level is bracketed by the interval defined by the cardinal three samples involved as analysed by seller, buyer, and umpire. But this assumption is challenged by the fact that the crucial primary sample (which is immediately divided in three sub-samples) is in fact the result of sampling a heterogeneous material. TOS-initiated readers will have smelled a scent already ....

4.3 Theory of Sampling (TOS)

All materials in the realm of technology, industry, processing, trading, commerce ... for which Testing, Inspection and Certification (TIC) is on the agenda, are heterogeneous – it is only a matter of degree. The Theory of Sampling (TOS) has for over 70 years demonstrated the severe danger involved in assuming that there is no sampling error involved extracting a primary sample. But this is not the place to detail TOS, as there is an ample source of excellent background literature available [3-8].

Without a modicum of TOS competence, the assay exchange scheme appears simple, easy-to-understand, and could not possible lead astray ...

4.4 Assay Exchange paradigm - the grim reality

However, the technical reality behind the Assay Exchange paradigm is grim, as shown in Fig. 1, which emphasises that no less than two sampling operations are involved, each with its own sampling/sub-sampling errors and uncertainties – and all this indeed takes place before analysis. Surely this cannot avoid having an impact on the quality, and as it transpires, the quantity reported in the analytical results.

Following TOS, when sampling heterogeneous materials (e.g. broken ores, aggregate materials, concentrates, mixtures, recyclates - in general materials with significant grain-size and/or density contrasts), there is every reason to take notice – and especially to take appropriate operational precautions regarding the impact of the dominant primary lot sampling error (PSE) [3-8].

And there are equally good reasons also to mind the subsequent 'sample division' (aka sample splitting) producing the three tacitly 'assumed equal' samples for the seller, the buyer, and the umpire laboratory (if needed). This sub-sampling is generally associated with a secondary, non-vanishing sampling error (SSE), the magnitude of which can be both small, significant or large, depending on the operational competence (and the material heterogeneity). Indeed, SSE may often be smaller than PSE, but never neglectable, especially if suitable practical facilities to be used for appropriate comminution (crushing) and mixing are not mandated in contractual stipulations to be TOS-compliant, which is often not the case [3-8]; see also the reflections on the Round Robin test framework at the end of this article.

The degree to which is has been possible to reduce PSE will determine how close the analytically determined concentration in the primary sample will be relative to the 'true' lot concentration. Following TOS' understanding there are always also secondary sampling errors (associated with sample splitting), although never as large as those associated with primary sampling. The primary sampling stage errors typically dominate the total uncertainty budget, and it is therefore always necessary to take the most stringent science-backed precautions at this stage. This is practically never an element in typical Assay Exchange agreements and in relevant trading contracts.

Appropriate precautions first and foremost include TOS' ability to eliminate the so-called Incorrect Sampling Errors (ISE), which is the necessary condition for unbiased sampling [3-8]. But this is necessary for all sampling stages, because any non-zero level of residual heterogeneity within a sample/sub-sample opens up for non-vanishing uncertainty contributions related to splitting operations. These trivial TOS insights are unfortunately most often unrecognised in the assay exchange realm, with severe consequences to be laid bare below.

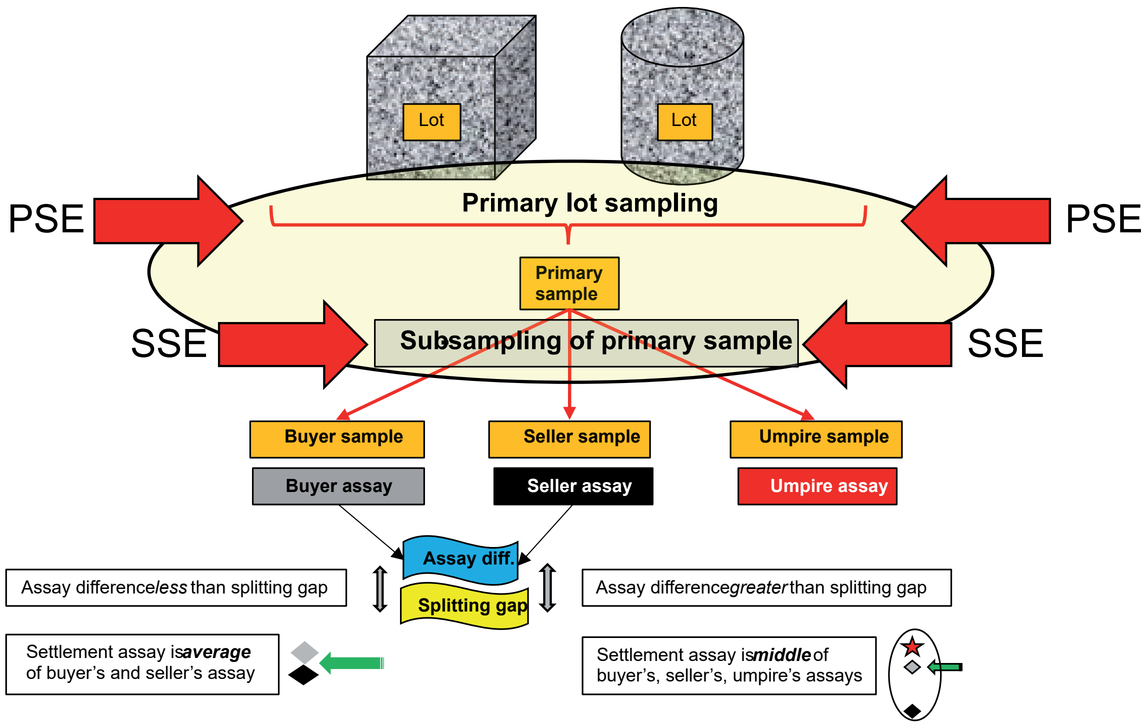


Figure 1: Assay Exchange reality – acknowledging two sampling stage uncertainty impacts PSE, SSE (red arrows). PSE := Primary Sampling Error; SSE := Secondary Sub-sampling Error. For full description of the Assay Exchange paradigm, see [1,2]. Illustration copyright: KHE Consulting teaching archive; used with permission.

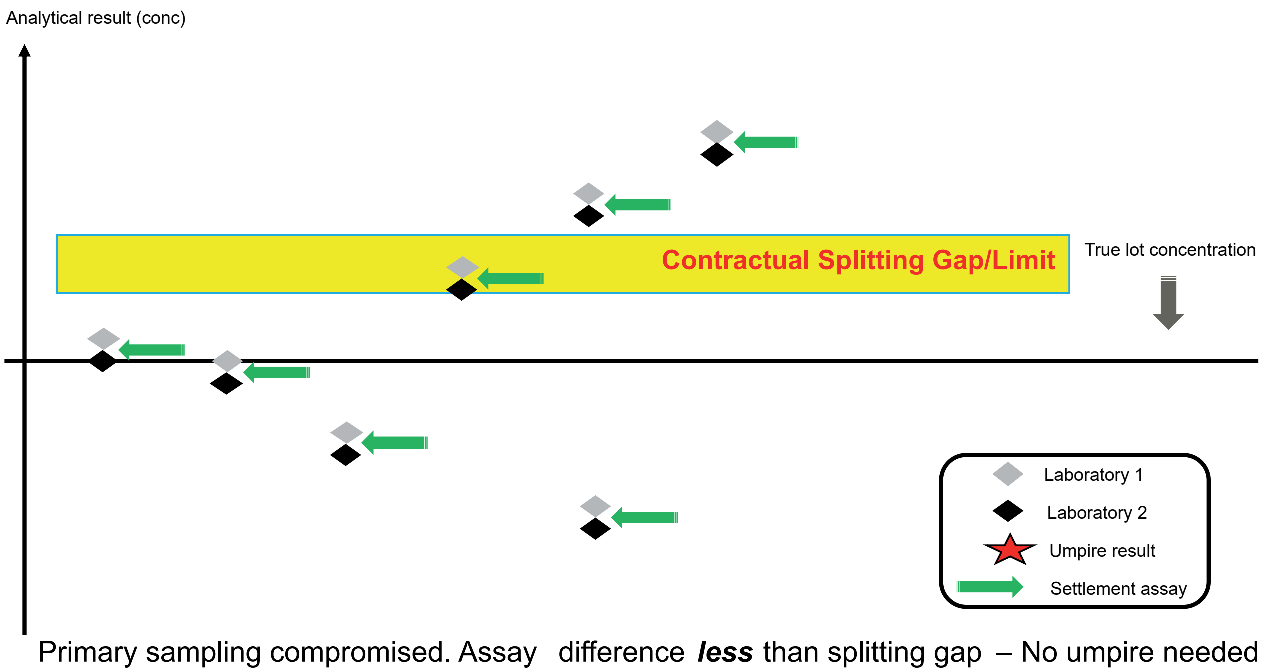


Figure 2: Illustration of a realistic splitting gap location (which is random as a function of the degree of incomplete sampling bias elimination and other potential ISE deficiencies at the primary sampling stage). The case illustrated here does not lead to 'go to umpire'; see [1,2] for full details. Illustration copyright: KHE Consulting teaching archive; used with permission.

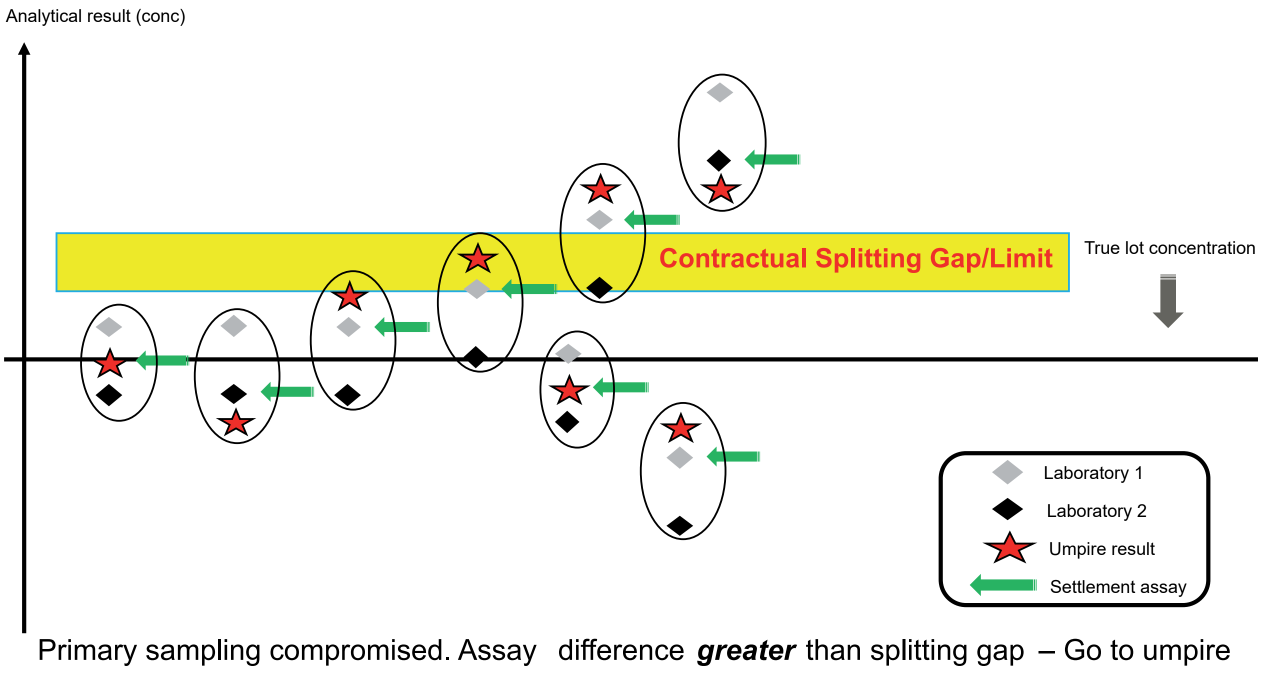


Figure 3: Illustration of a realistic splitting gap location (which is random as a function of the degree of incomplete sampling bias elimination and potential other ISE deficiencies at the primary sampling stage). The case illustrated leads to 'go to umpire'; see [1,2] for full details. Illustration copyright: KHE Consulting teaching archive; used with permission.

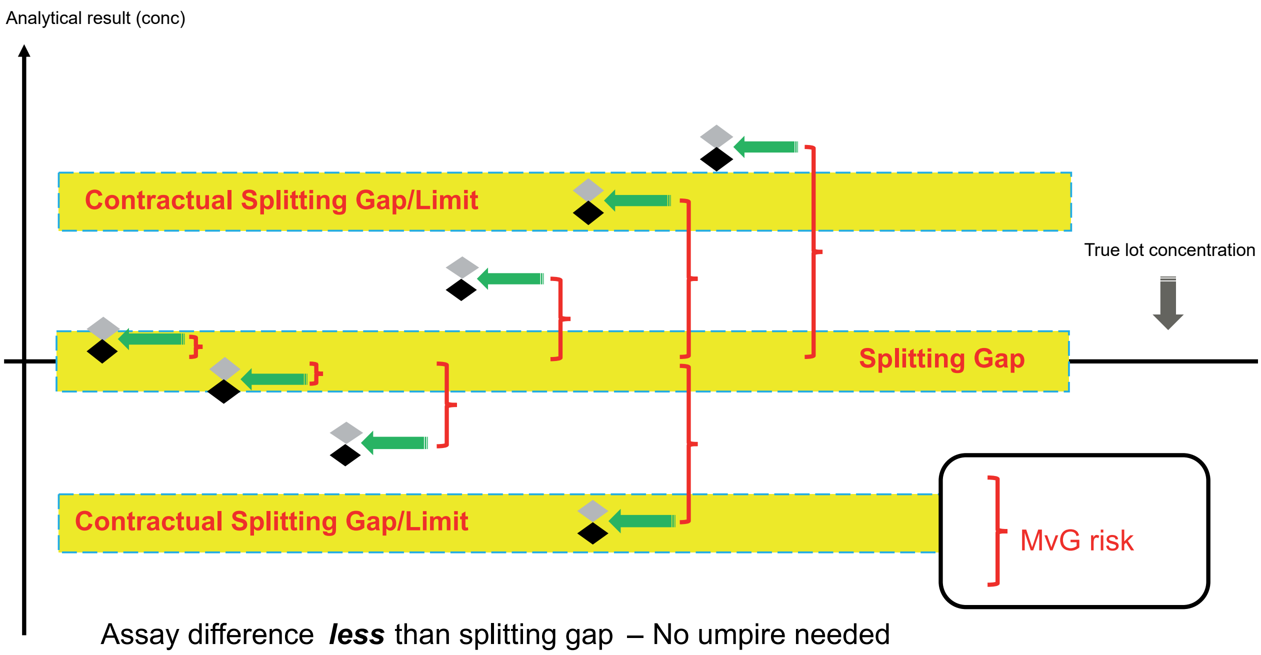


Figure 4: Illustrating the MvG risk; case of no umpire needed. The MvG error/risk is only (close to) zero in the case of vanishing primary sampling uncertainty; see [1,2] for full details. Illustration copyright: KHE Consulting teaching archive; used with permission.

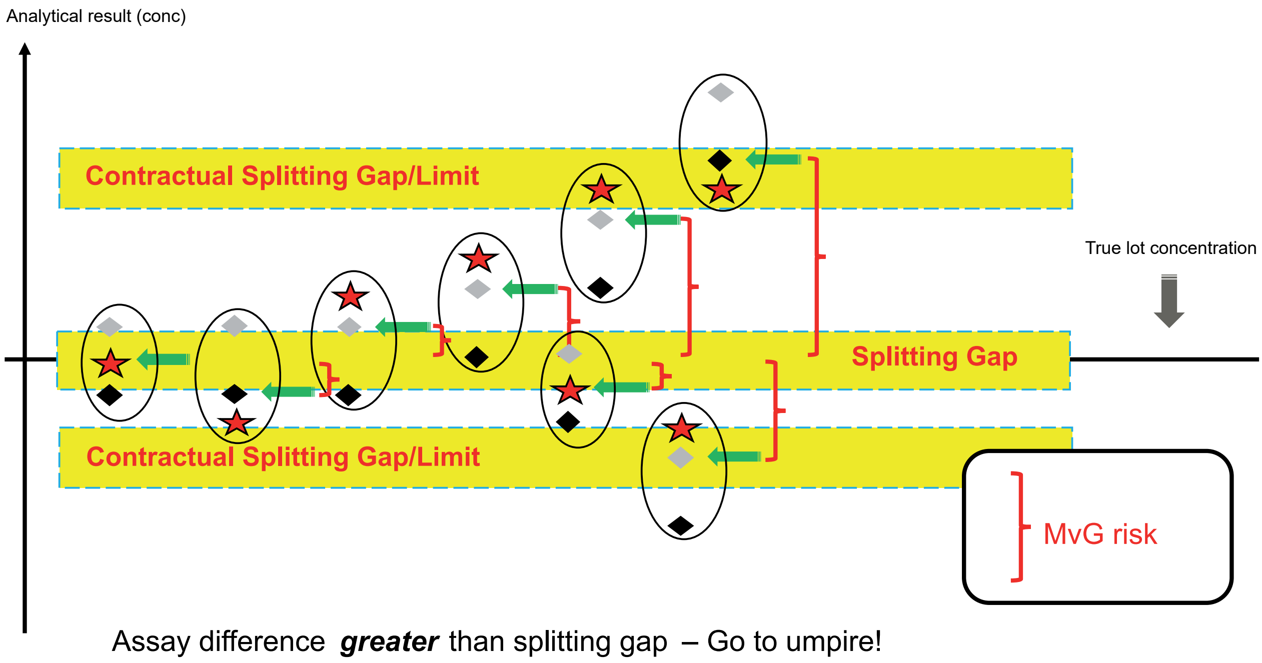


Figure 5: Illustrating the MvG risk; case of going to umpire. The MvG error/risk is only (close to) zero in the case of vanishing primary sampling uncertainty; see [1,2] for full details. Illustration copyright: KHE Consulting teaching archive; used with permission.

There are certain logical effects from the complementary PSE and SSE influences, as follows:

The degree of incomplete primary sampling bias elimination will impact the way the splitting gap is brought into action, manifested as a deviation from the assumed centering of the gap on the true lot concentration, as is shown in Fig. 2. This will be the situation regardless of whether the paradigm leads to 'go to umpire or not, Figs. 3-5.

It is important to emphasise the distinction between this primary sampling effect and the splitting gap mechanism. If the primary sampling uncertainty is nil, i.e. were it possible to eliminate all (incorrect – as well as correct) sampling errors (full explanation in [3-8]), the assay exchange paradigm would be unassailable. Were it possible to counteract all adverse effects from both material heterogeneity and from adverse sampling procedures (grab sampling in essence) and hereby always ending up with a perfect representative primary sample for all materials with all kinds and degrees of heterogeneity, always employing optimal composite sampling – then, and only then, would there be no splitting gap deviations.

Alas, the entire history of the Theory of Sampling (TOS) shows that this ideal scenario is not achievable under any circumstance! There is always a minimum material heterogeneity, giving rise to a non-vanishing minimum sampling uncertainty even after everything embedded in "best practice" has been adhered to. This is why one cannot glibly assume that the splitting gap is always centered on the true lot concentration, but rather that there will always be an offset to some degree (small, intermediate, large), witnessed in Figs. 2-5.

4.5 Mismatch vs. Gap error (MvG)

Any deviation between the final settlement assay value and the "true lot" concentration is termed the 'Mismatch vs. Gap' error, MvG [1,2], Fig. 5. This uncertainty translates into an economic risk, the MvG risk, which needs to be managed. However, this is demonstrably not a provision envisaged in the conventional assay exchange paradigm, which does not even acknowledge a primary sampling error (PSE) influence!

Adding in the subsequent sample division error effects (which may include a potential sub-sampling bias), the relative deviations between the three analytical results from seller, buyer, umpire is now crucial.

The tripartite assay results from Lab A, Lab B, and the Umpire lab depend on to which degree the primary within-sample heterogeneity has been successfully reduced/eliminated by appropriate TOS action before the practical sample division (primarily by careful mixing) which also contributes.

The full MvG risk can be illustrated with graphic clarity as shown in Figs. 4,5. There is here a subtle issue that needs to be emphasised with force:

Within the conventional assay exchange paradigm, this MvG uncertainty is always assumed to be zero; therefore, it's risk management never appears as a task on the business RM agenda.

Figs. 4,5 show how the location in the Y-axis direction of the contractual splitting gap may be significantly displaced from the assumed closeness to the true lot concentration, mostly influenced by the degree of a primary sampling bias if/when this has not been successfully mitigated [3-8].

Note that the assay exchange scheme is typically followed regardless of this critical uncertainty, an uncertainty which is not acknowledged in the standard assay exchange paradigm! Is this but an innocent oversight (bad when it comes to settlement), or is this deliberately overlooked? Or, worse, is this deliberately ignored? In all these cases, what could be the motivation for doing so? This harks back to the anonymous conversation with a TIC-agent quoted in the beginning of this article.

There is a substantial risk in not knowing about, or not acknowledging, the MvG uncertainty! The critical issue is that current assay exchange practices do not include mandatory means to deal with sampling influences in the splitting gap settlement accounting scheme! There are few exceptions from this situation which are associated with highly knowledgeable companies and corporations, or with entities who are interested in using this lack of understanding for narrow, selfish interests) .

4.6 The point is ...

Thus, there will always be a real, non-vanishing risk of settling a business transaction based on the assay exchange paradigm at an assumed reliable assay level, which may in fact lie significantly distanced from the true lot metal content, as demonstrated in Figs. 4,5.

The gap displacement shown here is a direct rendition of what in reality becomes a significant settlement bias, which is not known at the time of analytical determinations, and which therefore cannot be counteracted by any rational means.

The gap deviation, the MvG error/risk, may be small (low heterogeneity materials; largely acceptable sampling performance) but it may just as well be large (significantly-to-excessively heterogeneous materials; non-representative sampling competence/procedures/equipment):

The point is that all this is unknown to the contractual parties, who believe that the assay exchange scheme will result in a fair settlement for both parties.

For many commodities the economic risk involved may perhaps not constitute reasons for much worry (bulk commodities with relatively low unit value), but as tonnages go up, the sum-total economic effect may still well accrue to appreciable, unacceptable amounts ("The secret commodity trading club that runs the world"). This fact is much more characteristic in the mining, minerals processing and metal refining industry sectors. Here matters are economically much more serious, for example for the precious metals refining industry concerning e.g., Gold, Silver, Platinum Group Metals (PGM), some REE and similar, where even minute amounts of misassigned, but certified, deviating concentration results between buyer-seller translate into highly significant economic consequences – absolutely worth a legal fight if/when discovered. As an example, a misappropriation of one oz of Gold represents a value of USD ~4,500; one oz. of Platinum USD ~1,750; readers can do their own math.

Thus, there are many possible adverse consequences of not being aware of the MvG risk! It is precisely the scope, width and severity of such consequences that caught the attention of the accidental trio of reflecting gentlemen indicated in the introduction. It is not possible that these issues are not also known to many more parties with functioning eyes and an educated, rational mind. Which begs the question: How widely is this hidden risk known throughout the minerals and commodities trading world and in specific related industry sectors for miners, smelters, and refiners?

5. Manipulating mine and process metal accounting

There is another way reaching the same potentially adverse results – only here resulting from quite deliberate actions. For example, insights into how process industry laboratories have been coopted by local operational management to produce analytical data that improves desired metal accounting totals, which the specific processing industry is trying to achieve, or, in other words, actions which are deliberately trying to keep senior corporate management and shareholders 'happy'. This operational pressure can take many forms, but the most frequent one is probably refusing to accept analytical data not corresponding to expected or desired levels - instead continuing to re-analyse until a more fitting result is obtained by sheer happenstance (sampling, sub-sampling and analysis of heterogeneous materials will always result in some spread of analytical results) and then taking exactly this datum as gospel, rejecting all other data points as somehow flawed. If ever there was a more blatant verification that a total sampling + analysis variability is always present! This modus operandi is certainly not confined to ore and minerals processing and refining metallurgy industry sector alone!

The laboratory process disruption this behaviour causes affects the basic KPI's a laboratory manager and staff are measured against, i.e., negatively impacting turnaround, sample throughput, often prompting "client complaints", which then show up as negatives in performance reviews and, worst of all, reducing monetary performance bonuses. The result of this kind of pressure and financial penalties is that laboratory managers and senior staff may 'adjust' data before release to reduce, or completely eliminate re-analysis demands, to ensure a more "smooth laboratory process" with continued 'good' operational reviews. This practice is certainly not confined to industrial and commercial analytical laboratories only! Whenever there is a potential substantial extra value on the horizon, temptations abound.

Potential fraudulent behaviour specifically in the analytical laboratory aside, the focus interest in this article is how sampling and sub-sampling plays an integral role at many stages along the full 'lot-to-aliquot-to-analysis' pathway: Lots of temptations for failing one's way! There is an interesting testimony of a dramatic awakening to these issues by laboratory personnel with operational responsibility following a basic introduction to the Theory of Sampling (TOS) [10][3].

Other issues are associated with the many sampling and sub-sampling stages before analysis in general: industrial technical staff may be under pressure to take samples at 'advantageous times', to adjust sample size, or to 'modify' sampling procedures (including sample preparation), or to 'look away' at critical work path junctures - all again to provide final analytical results that will better support certain objectives, be these commercial, payment optimisation, metal accounting, trading profit, or environmental monitoring. This author has personally experienced the latter related to alleged 'random sampling' of spillage water on an oil production platform, where this kind of pressure was revealed when subsequently data analysing quarterly compositional results. The guaranteed loser in all such dishonest executions of sampling, sub-sampling, sample preparation and analysis is - representativity. When representativity is lost, all bets are off. A major ploy is how a simple physical tests and quantification like moisture determination can easily be used to bias analytical result in a favoured direction … by TIC inspection staff, by laboratory personnel, or by company or corporate management at different post-analysis levels.

Modified samples or process analytical samples/PAT sensor signals transformed into analytical data, will ultimately make it into data bases from which TIC companies and third-party laboratories will be tasked with providing independent analytical data to be used for final commercial settlements between i) buyer vs. seller; ii) seller's laboratory vs. buyer's ditto vs. the reference umpire laboratory – and so we're back to the assay exchange paradigm in many different (dis)guises, but with deliberately faulty, misleading, or downright fraudulent actions and data. What a world …

3 https://wcsb10.com/media/ogzbw012/wcsb10-economic-arguments-dec-2022-compressed.pdf (p.36)

6. Increasing profitability by 'tuning' sampling and assaying for exchange

The net effect of the above bad practices (example selection in no way exhaustive) is voluntarily to bias the assay exchange database, which will therefore in effect disadvantage one or more of the parties in a final commercial transaction. So ...

1. There can be involuntary, unrecognized bias-generation in the form of assay exchanges influenced by the hidden sampling error impacts described above: This is negligence by lack of understanding the full framework of assay exchange, treating it as but an always true and fair 'scheme' to be followed in a spreadsheet. Or
2. iThere exist various forms of deliberate manipulations at one or more stages along the 'lot-to-aliquot-to-analysis-to-risk management' pathway. While the former is a clear lack of professional due diligence, the latter reflect a direct fraudulent, criminal intent – but they both lead to the same result, an untrustworthy settlement/ transaction database.

There also exist other potential 'levers' that can be activated within the regimen of analysis alone, e.g. application of 'correction factors' of many kinds based on data whose origin is lost in a historical fog or deliberately kept hidden. But the analytical realm sensu stricto is not a topic for the present focus.

A final topic is associated with the field of risk mitigation practices, which has its root in a combination of asymmetric factors inherent in the formalisms of 'by the book' assay exchange and the ensuing settlement mechanics. This is where e.g., metal refiners seek to manage exposure to Measurement Uncertainty (MU). But is this MU := [MUanaysisl] or MU := [MUsampling + MUanalysis]? There is obviously a crucial difference! Industrial risk mitigation often involves anticipated, perceived, or misguided notions as to counterparty positioning. This is a complex mine field, driven by confidence in one's own superior ability to shine a torchlight through the dense fog of potential economic gains vs. loss understandings of what goes on following the Assay Exchange mechanics. This could manifest itself as a deliberate over–, or sometimes under-reporting analytical concentration values in anticipation of counterparty actions (which may or may not actually be the case) and/or even trying to factor in general umpire performance as well. Obviously, this kind of economic gaming can rapidly develop into rather byzantine risk management practices – but such practices are not on the agenda here.

What is important is that for some involved parties, as well as for some neutral observers, this economic game can sometimes seem to be so widespread that it takes on what could almost be viewed as a legitimate industry 'commercial practice'! But is it? The ultimate 'number manipulating' potential is so many-faceted that it needs its own comprehensive account (not touched upon here).

However, it is interesting how a key role is played out by sampling in this game: proper, representative sampling vs. sampling under duress.

So, what can be done? What should be done?

A preliminary step is to clear up the sometimes-confusing terminology used across different disciplines and applied technology fields: error vs. uncertainty vs. measurement uncertainty [11].

7. The way forward: Understand and eliminate sampling errors, uncertainties, risks

Status quo is that the standard assay exchange paradigm largely ignores the MvG risk.

The above analysis of misplaced, economically dangerous trust in the conventional Assay Exchange scheme without recognition of the two hidden sampling error uncertainty influences is tantamount to a significant unrecognized extra risk, almost never properly recognised. Embedding TOS errors (as TOS risks) in the general framework of Risk Management is treated in detail by Paoletti and Esbensen (2025) [12].

All the above complex technical relationships notwithstanding, there is a surprisingly simple way out of this under-brush of potentially nefarious behaviours:

It starts with the fundamental need for selection of a Decision Unit (DU); technical details can be found in [13]. The cardinal step in designing a representative sampling plan 'from-lot-to-aliquot-to-analysis' is a formal definition of an appropriate Decision Unit (DU), aligned with the transaction or settlement objective. The Decision Unit determines unequivocally the specific material target (in terms of location, timing, volume, mass) from which sample increments are extracted, as part of a composite sampling process [3-8].

The DU is the specific material to which the final analytical results apply and from which inference is to be drawn as to the characteristics of the whole lot. By insisting on an a priori DU definition, there can no longer be applied 'encouragement' or pressure to technical personnel, because there now exists a comprehensive, complete operational SOP (Standard Operation Procedure), which in the name of transparency of course is meant to be followed to the letter! Should 'deviations' later be discovered, the meticulously worked out DU definition, SOP and sampling plan will also serve to protect workers and personnel below supervising management levels in relation to criminal prosecution, lawsuits ...

8. Resolution

Here is the universal resolution – and it is an easy one. All that is needed to resolve the critical issues outlined above is 100%, stringent adherence to the principles of the Theory of Sampling (TOS) by mandatory contractual stipulations only to use (and only accept from counterparties) documentable TOS-representative sampling and sub-sampling practices; full details in [14,15]. As concerns the key sampling aspects, there is only need for the following two sentences that embodies the core of what shall be done:

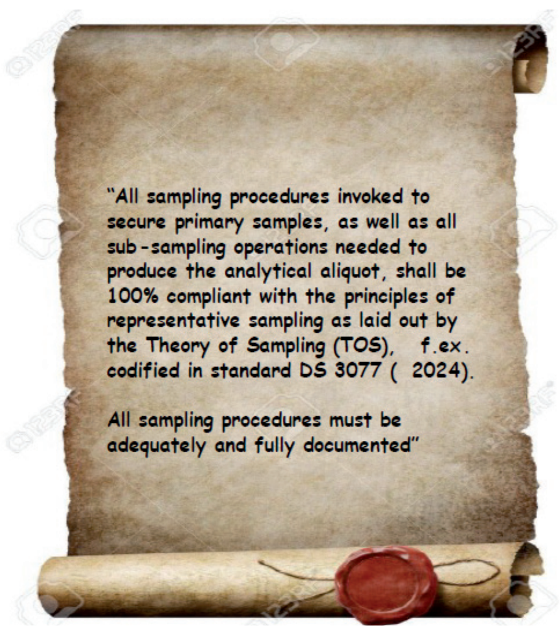


Figure 6: Contractual, settlement and agreement mandate – universal remedy against all sampling deficiencies. Illustration copyright: KHE Consulting teaching archive; brought here with permission.

Imagine the relief if/when the two sentences quoted in Fig. 6 were to be included in all trading and settlement contracts going forward. The entire array of debilitating issues regarding the assay exchange scheme - and all temptations to bad behaviour (re. sampling, analysis, risk management) could go away if/when this mandatory contractual stipulation is agreed upon by all parties!

Well, what is also needed is to instigate some directed business and cultural moral re-armament ...

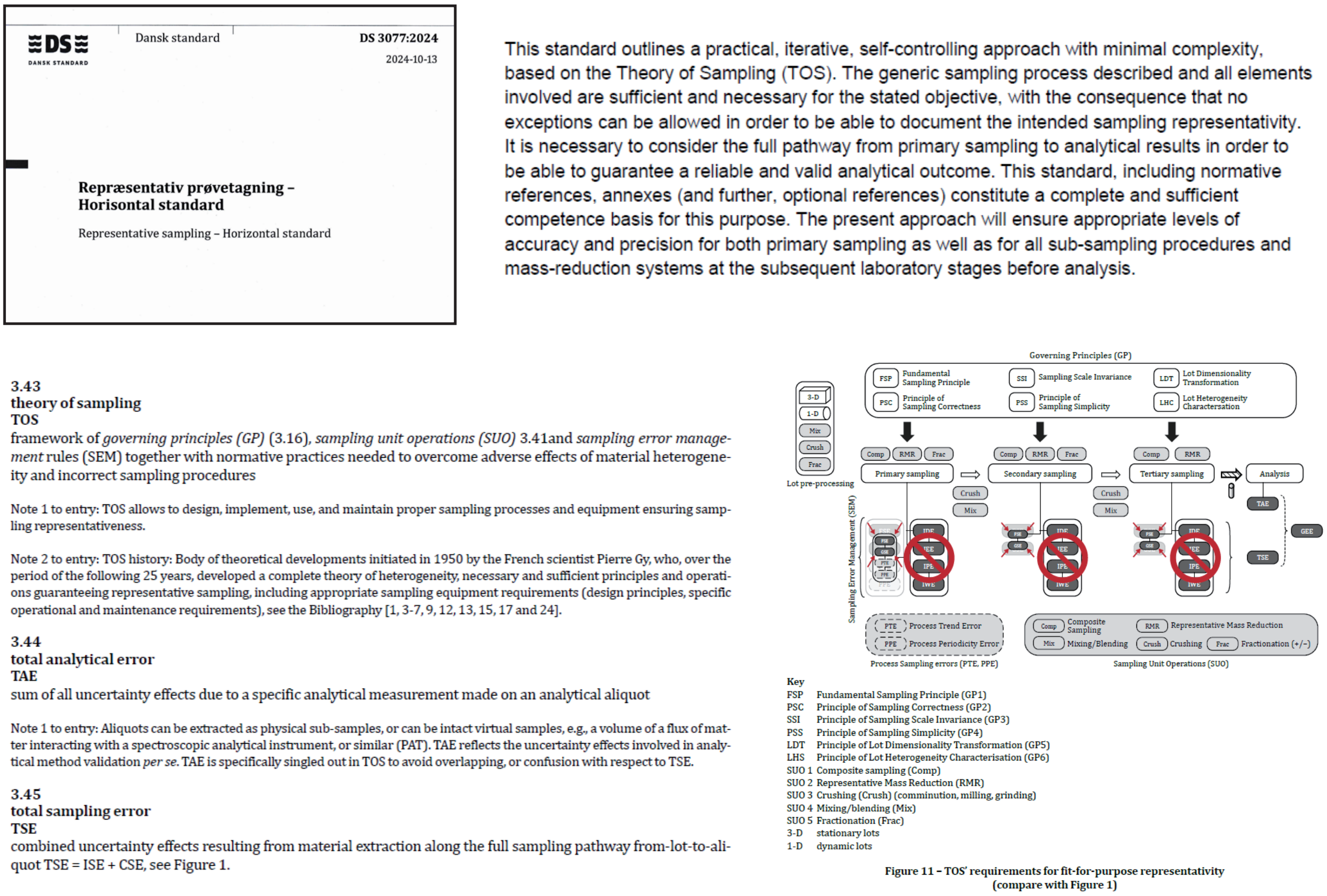
Then:

1. All sampling procedures would only be in the form of proper composite sampling (grab sampling is banned).
2. All bias-generating Incorrect Sampling Errors (ISE) would be eliminated from the primary sampling stage and from all sub-sequent sub-sampling stages as well, forcing MU:= [MUsampling + MUanalysis] to be optimally reduced to MU:= [MUanalysis].
3. All industry sectors where sampling is a critical element will henceforward be honour-bound to follow the global standard DS 3077:2024 [14,15], Fig. 7.

9. Round Robin – no validation

Various suggestions are often given in defence of the assay exchange paradigm, for example: "Samples should regularly be sent for Round Robin (RR) assaying and comparison". To which: The findings of RR comparisons, also called Proficiency Testing Programs, are highly sensitive to whether the sample division process producing assumed 'identical replicate samples' sent off to participating (and umpire laboratories) are indeed representative, or not - and are, as such, indeed scrupulously identical.

The Round Robin framework is based on the tacit premise that the distributed laboratory samples (which can be viewed as an augmented seller – buyer – umpire sample set), are indeed 100% identical. If/when not enough attention, or no attention at all, is directed at the critical dividing sub-sampling involved, the RR scheme is subject to the exact same critique as levelled above to the standard assay exchange mechanism. When no attention is directed to the sampling issues involved, the RR facility is only able to compare the analytical performances s.s. between laboratories (which is indeed the official objective of RR), but this leaves the sub-sampling variance out of consideration, effectively allowing a MvG risk/uncertainty to infiltrate the comparative analytical database.



Dansk standard DS 3077:2024

2024-10-13

Repræsentativ prøvetagning – Horisontal standard

Representative sampling – Horizontal standard

This standard outlines a practical, iterative, self-controlling approach with minimal complexity, based on the Theory of Sampling (TOS). The generic sampling process described and all elements involved are sufficient and necessary for the stated objective, with the consequence that no exceptions can be allowed in order to be able to document the intended sampling representativity. It is necessary to consider the full pathway from primary sampling to analytical results in order to be able to guarantee a reliable and valid analytical outcome. This standard, including normative references, annexes (and further, optional references) constitute a complete and sufficient competence basis for this purpose. The present approach will ensure appropriate levels of accuracy and precision for both primary sampling as well as for all sub-sampling procedures and mass-reduction systems at the subsequent laboratory stages before analysis.

3.43
theory of sampling
TOS
framework of *governing principles (GP)* (3.16), *sampling unit operations (SUO)* 3.41and *sampling error management* rules (SEM) together with normative practices needed to overcome adverse effects of material heterogeneity and incorrect sampling procedures

Note 1 to entry: TOS allows to design, implement, use, and maintain proper sampling processes and equipment ensuring sampling representativeness.

Note 2 to entry: TOS history: Body of theoretical developments initiated in 1950 by the French scientist Pierre Gy, who, over the period of the following 25 years, developed a complete theory of heterogeneity, necessary and sufficient principles and operations guaranteeing representative sampling, including appropriate sampling equipment requirements (design principles, specific operational and maintenance requirements), see the Bibliography [1, 3-7, 9, 12, 13, 15, 17 and 24].

3.44
total analytical error
TAE
sum of all uncertainty effects due to a specific analytical measurement made on an analytical aliquot

Note 1 to entry: Aliquots can be extracted as physical sub-samples, or can be intact virtual samples, e.g., a volume of a flux of matter interacting with a spectroscopic analytical instrument, or similar (PAT). TAE reflects the uncertainty effects involved in analytical method validation *per se*. TAE is specifically singled out in TOS to avoid overlapping, or confusion with respect to TSE.

3.45
total sampling error
TSE
combined uncertainty effects resulting from material extraction along the full sampling pathway from-lot-to-aliquot TSE = ISE + CSE, see Figure 1.

Key
FSP Fundamental Sampling Principle (GP1)
PSC Principle of Sampling Correctness (GP2)
SSI Principle of Sampling Scale Invariance (GP3)
PSS Principle of Sampling Simplicity (GP4)
LDT Principle of Lot Dimensionality Transformation (GP5)
LHS Principle of Lot Heterogeneity Characterisation (GP6)
SUO 1 Composite sampling (Comp)
SUO 2 Representative Mass Reduction (RMR)
SUO 3 Crushing (Crush) (comminution, milling, grinding)
SUO 4 Mixing/blending (Mix)
SUO 5 Fractionation (Frac)
3-D stationary lots
1-D dynamic lots

Figure 11 – TOS' requirements for fit-for-purpose representativity (compare with Figure 1)

Figure 7: DS 3077:2024 "Representative Sampling: Horisontal standard". Global standard stipulating the necessary-and-sufficient framework for defensible sampling in all of science, technology, industry, environmental monitoring, food, feed, pharmaceutical manufacturing/processing, regulatory authorities [14,15]. Illustration copyright: KHE Consulting teaching archive; brought here with permission.

Of course, conscientious RR organisers recognise that potential errors in subsampling may significantly influence the intended statistical comparison of laboratory analytical data, impacting on the validity of the conclusions on performance of the participating laboratories. Therefore, RR samples are often prepared to a much smaller particle size than what is common for commercial settlement samples and then subjected to (very) thorough mixing. One often stated reason is that a much-reduced particle size is helpful since it will increase mixing efficiency and will also help to reduce extraction errors e.g., from smearing gold left behind on the pulverising instrument, or by discarding oversize sample material that is too hard to crush. With RR this intensive sample preparation 'does not matter' as the aim is to compare analytical performances only. An RR dispatching facility will sometimes perform perfunctory homogeneity checks to verify that each divided sample generates the same analytical result when analysed with its own in-house laboratory. Only after such vigorous measures have satisfied the dispatching facility, will it send out the RR test samples to participating laboratories.

Notably however, such proficiency test samples no longer have a defensible representativity relationship with the donor material from which they originated; they have specifically been prepared to make the RR exercise, or Proficiency Testing Program, only, focusing on the relative analytical performances. It is as if sampling and especially sub-sampling effects in this case do not exist and need not be taken into consideration. But they most certainly do exist and will most definitely impact the comparison database; it is just that nobody cares about with which influences and magnitudes that have been left out of consideration. These sampling/sub-sampling effects will be incorporated in various in-between sample and in-between laboratory performance variances.

These 'optimised sample set' issues for RR purposes are in fact identical to the standard assay exchange setup in that the relationship to the original lot composition will never be known.

While this may be fully acceptable in the case in which one is well-knowingly, deliberately only interested in analytical performance comparison (which indeed is the raison d'etre for RR), this also means that there is no saving grace w.r.t. the assay exchange paradigm even if samples are "regularly sent round for Round Robin (RR) assaying and comparison". RR checks will never be able to detect and to quantify the associated sub-sampling errors and their resulting uncertainties. Therefore, to try to make RR testing more relevant, more realistic, efforts have been expended by some RR test organisers instead to employ real-world samples for the RR comparison of analytical performance, which are then subjected to Herculean mixing efforts a.o. before splitting. It is easy to understand the rationale for this based on the present exposé: Analytical laboratories are supposed to be able to analyse original lot material with optimal accuracy and precision (field samples, industrial samples, trading samples, environmental samples ... target materials are very different in nature). However, accuracy in the RR context is exclusively w.r.t. the analytical aliquot volume only. But this context is identical with the assay exchange context treated here. Thus, this more 'realistic' RR context will be subject to the exact same critique as was levelled at the standard assay exchange scheme above, although often with significantly reduced splitting uncertainties. Principally however, the result is that this version of the RR facility is equally unable to serve as validation the assay exchange paradigm.

Summing up: Agreement between analytical results from the same laboratory only attests to the consistency of analysis, while agreement between different laboratories does not guarantee representativity with respect to the original lot material. 'True lot concentration' cannot be measured - which is why the Theory and Practice of Sampling (TOS) was developed; see [8, Fig. 8] for an overview of the historical development of sampling practice and theory as solutions to societal needs and demands. Systematic effects will always arise when there are differences in i) sampling procedure, ii) preparation and iii) analytical techniques.

So, even the most analytically precise assay values are only the best possible estimates of the compositional characteristics of an original lot, subject to both random variability and potential systematic effects, reflecting the compound, complex, multistep sampling pathway from lot to aliquot — all of which takes place before analysis. And then there is risk mitigation, a game of its own, sadly not exclusively performed by former boy scouts ...

10. Conclusions

This contribution offers the dictum presented in Fig. 6 to all sectors in science, technology, industry, trading, commerce, regulating authorities, environmental monitoring and control, risk management (exemplification nor exhaustive) where sampling plays an integral role for reliable, representative analytical results upon which to be able make transparent, defensible decisions in society - together with a modicum of moral re-armament to fight temptations to economically selfish narrow-term interests!

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The author acknowledges revealing discussions leading to key insights into the murkier aspects of contractual settlement and commercial practices (overt, as well as covert) with key colleagues and industry representatives, who, for reasons not overly difficult to understand prefer to remain undisclosed. I thank them profusely for their valuable help to lay bare some of the behaviours described here — they know who they are. Two reviewers helped tremendously toning down and taking the edge of some of the remarks in the original manuscript - also greatly appreciated.

FURTHER READING

April 7th, 2026, the beginnings of a broader discussion of these issues could be found on LinkedIn under the heading "But let's talk about what actually happens on the ground". This discussion is a perfect complement to the present essay, greatly recommended

https://www.linkedin.com/feed/update/urn:li:activity:7447296871074525185/

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