SAMPLING COLUMN

# Representative mass reduction in the laboratory: riffle splitting galore (with or without errors)

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**While this series is presenting the universal principles behind representative sampling of all types of lots and composition, the focus has studiously been kept outside the analytical laboratory. This is because many are of the opinion that applying the Theory of Sampling (TOS) at all such large(r) scales (primary sampling) is different from the work thought to belong the analytical realm, which indeed takes place at much smaller scales. However, if the systematics of TOS shall be in a position to be used to its full power and reach, this division needs careful attention—it is time to enter THE LAB (see also the last column of 2016[1]).**

## Introduction

Truth be told, for the many operations falling under the term "sample processing" or "sample preparation", very nearly all contain straight-forward sampling processes—only **writ small**, but *bona fide* TOS operations nevertheless. As is shown below, it pays well to follow TOS' universal application scope all the way to its ultimate stage, that of selecting (sampling for) the analytical aliquot (the analytical mass). It is very advantageous to view all sampling operations, spanning the entire "from lot-to-analysis" pathway, as a scale-invariant theatre; in which the sampling operations are identical, in principle as well as in practice. It is indeed **only** the scale that varies. Thus a spatula – is a laboratory spoon – is a shovel – is a spade – is a backhoe grabber – is a crane grabber… All these tools are used to select and extricate an increment, or a sample, it is only the **scale** that varies. The choice of which sampling tool dimension to choose is only related to the lot size vs the desired increment size, all of which is strongly related to the grain size characteristics of the lot. The objective of collecting an increment, or several, is in practice always related to only two possible objectives: to perform grab sampling **or** composite sampling (see previous columns). The last column of 2016[1] dealt with some of these systematics in detail from the perspective of a particularly popular tool, the sampling spear, or the sampling thief. Following directly this avenue, the present column deals exclusively with the by far most often used method for mass reduction in the lab—riffle splitting.

## Riffle splitting

There are a few requirements in order for riffle splitting to be the perfect way to do mass reduction in the lab, by which is meant the most effective way to obtain representative mass reduction in the lab. The sample material must be free-flowing in order to be able to pass through the riffles, driven by gravity. Other than that, there are obvious requirements related to the largest particle size (in some less frequent cases also related to the sorting of the material). In general, it is obvious whether a target material is suitable for riffle splitting or not. It is the largest particle size that determines the operative requirements of the riffle splitters. A well-known rule of thumb is that the individual riffle opening must be three times the largest particle diameter + ε, in order to prevent all possibilities of *clogging* a riffle chute. With these few requirements in place, riffle splitting is completely *scale-invariant*, and one may pick the splitter tool that fits the practical and logistical conditions and requirements best, see Figure 1.

The riffle splitting principle can be implemented in a great variety of scales and ways, and realised in a wide range of tools, but the principle behind all is intuitively simple and easy to comprehend: the objective is to split an incoming mass into two *equal* sub-samples both with respect to mass and (which is decidedly most important) with respect to the analyte concentration to be found in each. There also exist variations aimed at different splitting ratios, see further below. The universal riffle splitting principle is illustrated in Figure 2.

Perhaps surprising, it is fully possible to conduct riffle splitting in a non-representative manner. Thus there are rules governing riffle splitting if this is to be representative. Below is illustrated some of these as but a first foray into the subject. For complete coverage of

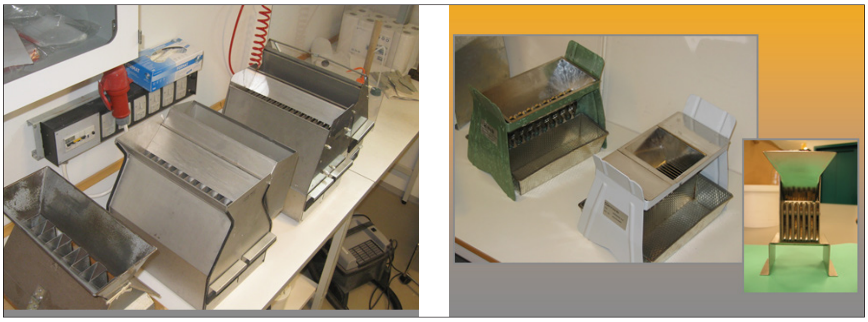

**Figure 1.** Size does not matter. Riffle splitters are available in a large range of sizes, determined by the effective opening of the riffles (chutes). The smallest met with so far is illustrated on the far right, managing to compress 14 juxtaposed chutes along a linear distance of only 5 cm. The resulting chute width is just about the smallest opening that can accommodate very fine grained aggregate material and powders without a serious danger of clogging. So riffle splitters smaller than this are not relevant, and other ways must be found (other tools) that manage to do sub-sampling in a fashion that achieves the same purpose.

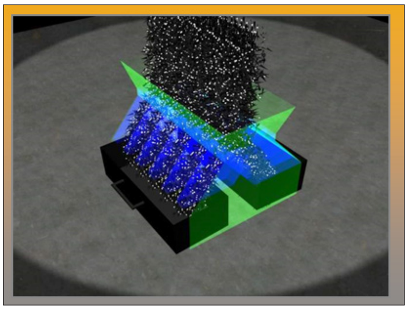

**Figure 2.** The universal riffle splitting principle: a collimated stream of matter is split by a series of juxtaposed riffles (chutes) leading to a number of slices of the stream into two alternative sub-sample reservoirs.

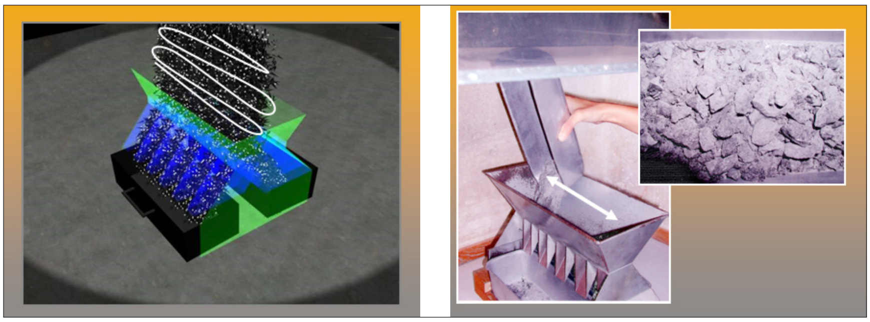

**Figure 3.** Longitudinal loading of the ingoing sample to be split is often an area of major misunderstanding. This "covering all chutes evenly" operation may well seem fair and reasonable at first sight, which upon scrutiny is revealed to be based on a faulty, undocumented, indeed unjustified assumption that the material in the loading tray is fully homogenous. As is very well known from TOS (see all previous columns), this never occurs in the world of science, technology and industry, and will always result in an unnecessarily inflated TSE.[4]

this critically important curriculum, see Petersen *et al.*[2] or Pitard,[3] but these basic issues are also covered in many of the background TOS literature references, see, for example, in the standard DS 3077.[4]

There is always a danger of that *some* of the component particles may accidentally bounce and rebound upon contact with chute walls etc. and thus may, accidentally, be propelled out of the active chute splitting zone. Such components are *lost* from the splitting products, i.e. an Incorrect Extraction Error (IEE) has been committed (by a structural Incorrect Delineation Error, IDE). Figure 5 illustrates why riffle splitters always must be closed or encapsulated. This is not too much to demand from any manufacturer.

In the right-hand image of Figure 5, a serious effort has been made to prepare the loading tray so as to deliver all the material along the longitudinal splitter axis in a controlled, even fashion (see also Figure 6). This, combined with the

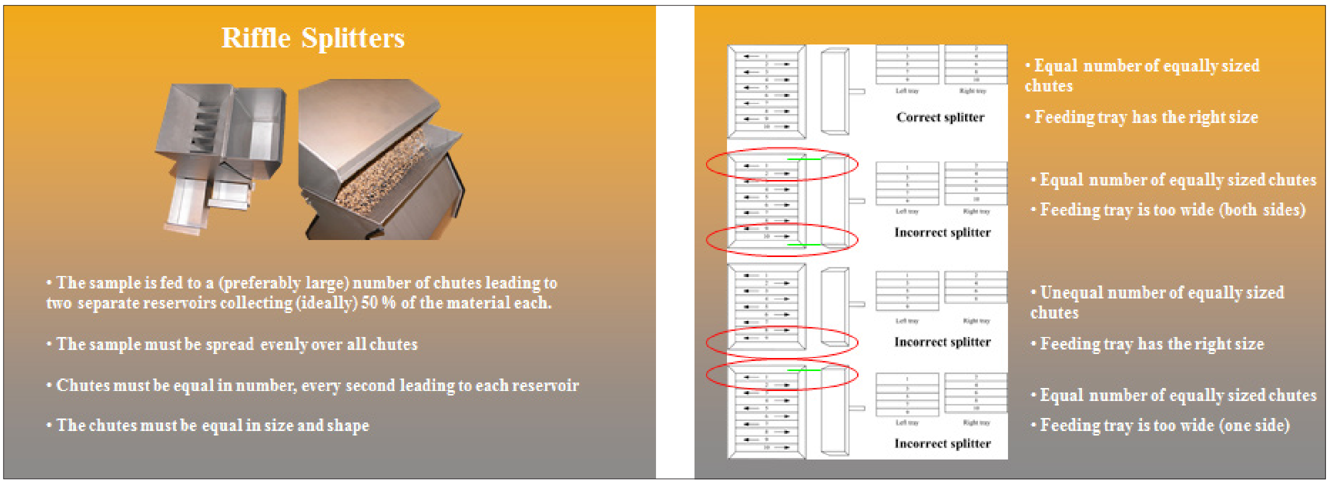


**Figure 4.** Left: riffle splitter design principles that must be observed. Right: the many ways to break these rules (most often unknowingly). However, it is the easiest thing to become an expert in all matters riffle-splitting.[2–4]

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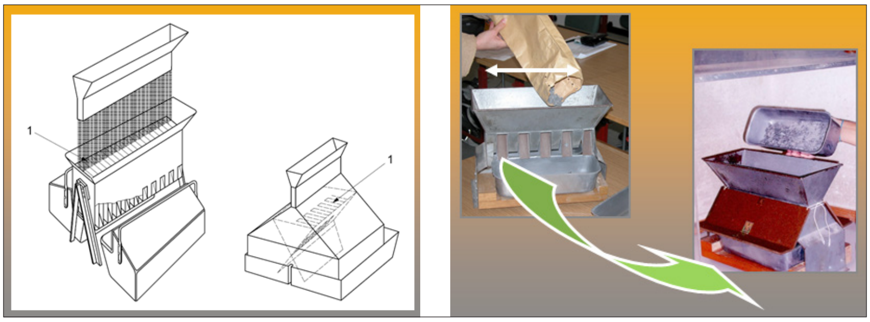


**Figure 5.** Left: how the closed equipment requirement can be easily realised, or **not**. Right: how the misunderstood "covering" loading is replaced by a carefully prepared loading tray being used so as to deliver all the material along the longitudinal splitter axis simultaneously in a controlled, even fashion. There has also been a serious effort to mix the material in the tray thoroughly before loading. All such operations help!

Figure 7 shows how a modest improved comminution results in a significantly improved loading tray material constitution, much better suited for improved splitting efficiency.

It is in the *interaction* between optimised material constitution and the number of riffle chutes brought to bear that riffle splitting mass reduction really comes to the fore—with a significantly improved (i.e. reduced) TSE. In Figures 8 and 9, a very heterogeneous, very unevenly distributed material (almost a caricature) in the loading tray (red/black)

**Figure 6.** Albeit using only primitive and simplistic prototypes, in this analytical laboratory, the riffle splitting operations shown do everything correctly, indeed in a representative fashion: no IDE, IEE because of correct pre-loading mixing, correct loading, using a correct enclosed splitter. And should there be a residual dust fraction escaping the operations (hopefully reduced to the absolute fit-for-purpose level), a plexiglass hood has been installed (operated by a powerful exhaust fan) taking care of health risks to workers.

critical effort to mix the material in the tray thoroughly, results in subsequent sampling (splitting) procedures with a significant reduction in both Incorrect as well as Correct Sampling Errors (ISE, CSE).

The above precautions can always be observed, it is only a matter of TOS meeting with GLP, so, many, repetitive mass reduction operations can be carried out even in quite extensive scales with only small efforts (there is always relevant equipment to be had).

As an example with great carrying-over effect: more thorough crushing is a very effective sampling unit operation that can be used with significant effect.

**Figure 7.** More efficient crushing leads to a much more uniform material in the loading tray, especially when combined with a conscious effort for better mixing as well.

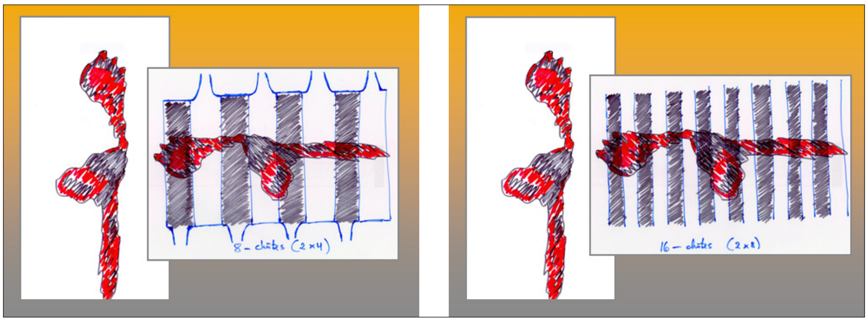

**Figure 8.** Illustration of a very sloppily prepared, extremely inhomogeneous material laid up in a loading tray (red/black), and subjected to an increasing number of active splitting chutes.

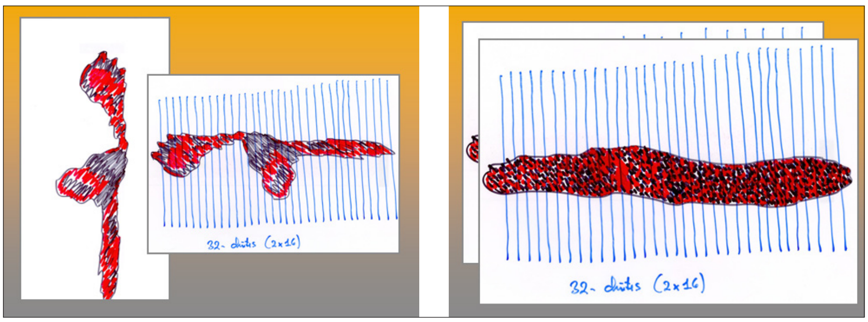

**Figure 9.** Left: illustration of a very sloppily prepared, extremely inhomogeneous material in a loading tray (red/black) subjected to a very high number of active splitting chutes. Right: an identical number of chutes, splitting the same material that has been subjected to **proper** crushing and mixing unit operations; this also increases the splitting efficiency significantly.

is subjected to a series of different splitting chutes (8, 16, 32), making it obvious that an increased number of chutes always offers better sub-sampling, everything else being equal. The last illustration shows the situation in which the most effective splitter (32 chutes) is brought to bear on a much improved material constitution (much better crushed and very well mixed material). The essential feature is that it is the exact same material subjected to four very different riffle splitting operations. There is absolutely no doubt that when crushing, mixing and effective riffle splitting are brought together with a well-considered, TOS-informed plan pertaining to the material characteristics at hand, the largest reduction in TSE can be obtained almost at no extra effort.

Observe how proper riffle splitting (using, say, *G* chutes) acts like a very through composite sampling—each of the two identical sub-samples were constructed by aggregating *G*/2 increments covering the entire lot (the ingoing load sample). This composite sampling effect is not always recognised.

Figures 8 and 9 clearly illustrate the advantages obtainable when calling in three of the four Sampling Unit Operations (SUO) in their right order (crushing, mixing, composite sampling) leading to the most efficient (least TSE) mass reduction possible in the analytical laboratory. Compare this to the plethora of sub-optimal, indeed often fatal, applications of grab sampling which can be observed in many of the world's laboratories in which the spatula still rules (see also the last Sampling Column of 2016[1]).

## Automation—enter the rotary divider

If not already, at one time or other, the advantages of using riffle splitters for effective TOS-correct mass reduction will become obvious, indeed pressing. All the necessary, but repetitive, manual work will at first be a blessing because of the dramatically reduced TSE involved. Soon, however, *all* this work will begin to look like a burden—"if only this work could be *automated*…".

Well, no problem: enter the *rotary divider*. Rotary dividers act and function precisely *like* a riffle splitter, in fact they *are* riffle splitters through and through, only designed for a much more efficient *throughput*. Figure 10 shows two versions of the rotary divider, one with fixed opening widths for the number of chutes chosen (32), and one with a variable chute width for the number of chutes chosen (12). For both there are now no limitations regarding the weight of the sample to be loaded, because any (large) sample mass can be loaded in successive parts without changing the sum-total splitting operation; this is a huge advantage both for the high-throughput laboratory as well as with respect to on-line process implementation. Both the rotary dividers shown here operate on the basis of the same framework with a loading hopper and a rotating nozzle that delivers a steady stream of material hitting the splitting chutes which are arranged in a circular fashion.

By carefully balancing the loading flux in relation to the rotating nozzle speed it is simple to arrange for the sample mass to be split and distributed over a very large number of chutes; every new 360° turn of the nozzle allows the stream flux to be distributed over a new multiple of the fixed number of chutes (here 32, 64, 96 …); the number of operative chutes multiplication factor is staggering, making rotary dividers very much more efficient compared to their stationary, linear cousins. There are many other advantages associated with rotary dividers, see References 2–4.

It is fair to say that many other types of implementation of the same rotary splitting principle can be found; some of these will be covered when this column turns its attention to process sampling.

## Benchmark study

There are an almost infinite set of variations on the theme of laboratory mass reduction approaches and methods, which type of equipment to use etc. Upon scrutiny and reflection, however, there are only a limited number of *types* of procedural approaches: grab sampling (spatula, spoon etc.), riffle splitting (linear, rotary), coning-and-quartering… A little systematics will clear the way for clear appreciation.

Figure 11 presents a graphical overview of the gamut of what is being used today in science, technology and industry laboratories—starting with grab sampling, i.e. using *one* extraction to get the analytical mass directly (TOS: obviously fatally wrong if/when homogeneity has not been documented beyond reasonable doubt), via "shovelling methods" with various fractional shov-


**Figure 10.** The rotary divider—the ultimate mass reduction equipment. Many variants of this principal solution can be automated.

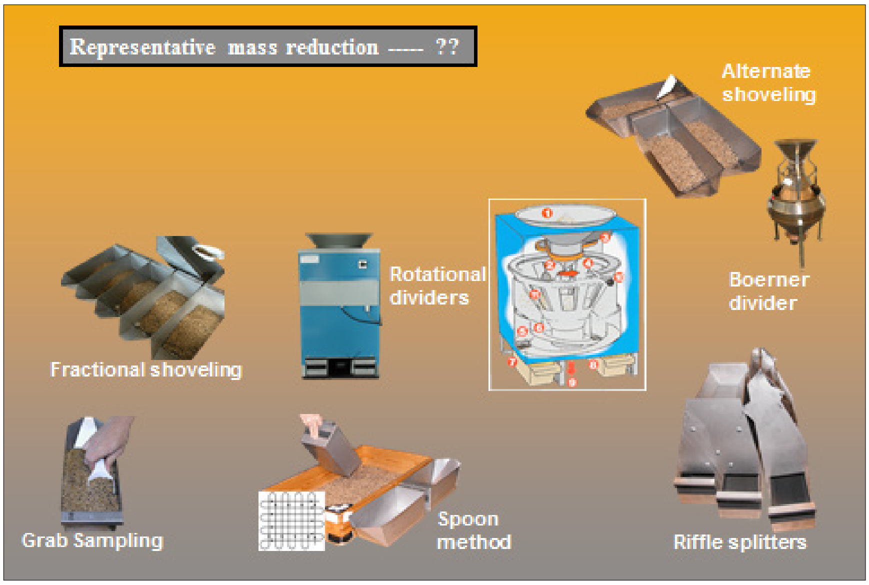


**Figure 11.** Overview of the principally different mass reduction methods and typical types of equipment in the authoritative benchmark study by Petersen *et al.*[2]

elling ratios (akin to simplistic composite sampling), to the well-known "spoon method" (used extensively in the seed industry) and the "Boerner divider" (a well-nigh brilliant invention from the same realm, Figure 12), to linear as well as rotary dividers.

And then there is *coning-and-quartering*, which turns out to be the world's most misunderstood combination of inferior mixing followed by a fatal four-riffle splitting—to be avoided at all costs. Coning-and-quartering (C&Q) was treated in full detail in a paper that could have had the title: "Why we killed C&Q and why it had it coming"—but which has a more scientifically acceptable title (with the exact same content, however), see Reference 5 for the full story.

At the other extreme is the "Boerner divider", which is named after its designer, Herr Professor Doktor Boerner (no doubt as to the nationality of its inventor). The functional principle is gravity driven, azimuthal cone-dispersion, sectorial chute splitting (34 chutes) without any moving parts. The principle is sheer genius, and is illustrated briefly in Figure 12. Better still "Look it up, look it up – *Google it*". Not only is the design brilliant, its appearance is often also a thing of beauty (such balanced use of brass and copper).

## The ultimate method/equipment ranking for the laboratory

The present foray through the typical objectives, methods, equipment design and means-of-operation for mass reduction in the laboratory has been swift, but manages to be comprehensive. In fact, all the principal types of mass reduction methods used in today's laboratories in science, technology and industry are covered, as are their typical practical manifestations with a necessary focus on "how to perform wrongly" (there is so much to learn, and to learn most effectively, from *mistakes*).

Figure 13 is the summary *representativity ranking* of all methods and approaches.

From TOS', from Referene 2, definition of representativity: $r^2 = (\text{bias})^2 + (\text{imprecision})^2$ suffice to say that the *smaller* the $r^2$ the *better* the sampling, i.e. the splitting approach/method/equipment! Detailed scrutiny of the plot reveals the general conclusions of this extensive benchmark:

- shovelling methods off all kinds are *unacceptable* (excessive TSE, excessive $r^2$);
- the riffle splitting *principle* reigns supreme, rotary over linear when possible, but both variants works exceedingly well—critically dependent on proper eradication/reduction of all ISE, CSE);
- the "Boerner divider" is superior to pretty much anything else.

**Figure 12.** The famous "Boerner divider", functioning exactly like a rotary divider but **without** moving parts. Every second chute leads to two separated collecting conical funnels (inner and other), allowing complete separation into two identical sub-samples.

## Conclusions

So, mass reduction in the laboratory is anything but the easy matter of acquiring a piece of equipment that *claims* to be able to do a representative splitting job. Far from it: performance documentation is needed! Well there is one exception, which unfortunately cannot be applied to all types of material, but

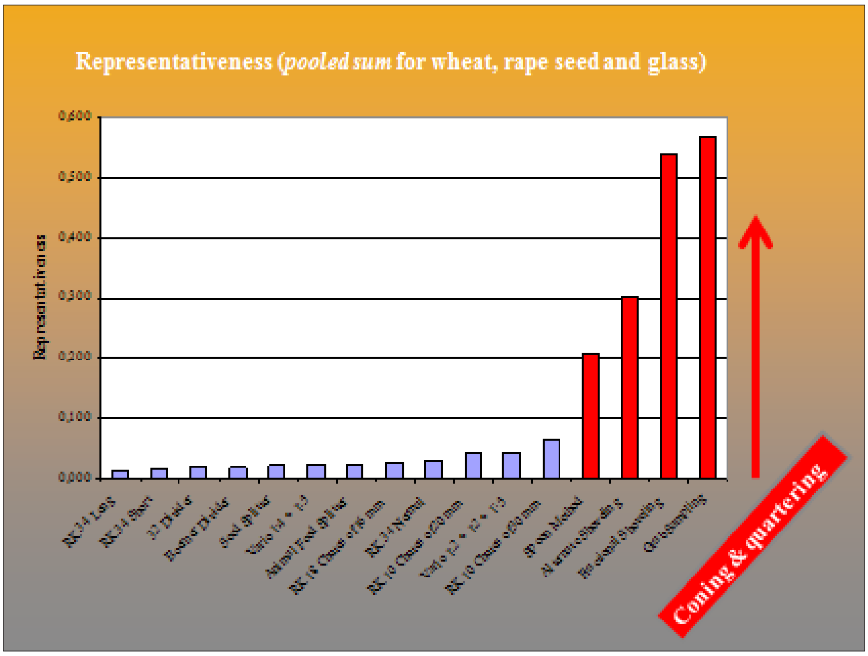


**Figure 13.** Ultimate representativity ranking of the 17 + 1 methods assessed in Reference 2. (The coning-and-quartering method was added after publication.[5])

when this is the case, just order the Boerner divider ;-)

For all types of equipment that have passed muster in the representativity ranking[2] there exists a rational set of rules that *must* be honoured in full in order for any alleged "splitter" to be representative. The most important of these have been introduced and illustrated above. An authoritative benchmark study allows anybody to perform a comprehensive audit of the state of TOS application in the analytical laboratory, greatly recommended.[2,3] A severe warning is sounded about coning & quartering,[5] incidentally at all scales.

## References

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