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Representative mass reduction in sampling—a critical survey of techniques and hardware

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Abstract

We here present a comprehensive survey of current mass reduction principles and hardware available in the current market. We conduct a rigorous comparison study of the performance of 17 field and/or laboratory instruments or methods which are quantitatively characterized (and ranked) for accuracy (bias), reproducibility (precision), material loss (external as well as internal loss), user-dependency, operation time, and ease of cleaning. Graphical comparison of these quantitative results allow a complete overview of the relative strengths and weaknesses of riffle splitters, various rotational dividers, the Boerner Divider, the "spoon method", alternate/fractional shoveling and grab sampling.

Only devices based on riffle splitting principles (static or rotational) passes the ultimate representativity test (with minor, but significant relative differences). Grab sampling, the overwhelmingly most often used mass reduction method, performs appallingly—its use must be discontinued (with the singular exception for completely homogenized fine powders). Only proper mass reduction (i.e. carried out in complete compliance with all appropriate design principles, maintenance and cleaning rules) can always be representative in the full Theory of Sampling (TOS) sense. This survey also allows empirical verification of the merits of the famous "Gy's formula" for order-of-magnitude estimation of the Fundamental Sampling Error (FSE).

Keywords: Mass reduction; Sampling; Riffle splitter; Shoveling; Boerner Divider; Rotational divider; Grab sampling; Representativeness

Sampling is nothing but representative mass reduction. [Pierre Gy]

1. Introduction

The archetype error of ill-reflected sampling is to focus on getting to the final sample volume much too early in the sampling process. Instead of only focusing on securing as quickly as possible the desired representative samples (which cannot be evidenced from the physical samples themselves) of the final sample volume/mass, the Theory of Sampling (TOS) stipulates that only a properly designed and controlled sampling process can facilitate this. Only TOS tells comprehensively how and how much material to extract from a lot. For many types of heterogeneous material often the extracted primary sample has to be of a quite substantial size in order to be representative, and this places stringent demands on the sampler (the sampling instrument), for instance if the sample is used for chemical analysis, where typically only 1 g, or a fraction hereof is required. Usually, there is a very long way from the size of the initial lot—via the primary sample—to the final analytical sample mass (Fig. 1). Typically mass reductions of the order of 1:1000 to 1:100.000 have to be invoked. It is therefore of the utmost importance that all sampling processes make do only with representative mass reduction. Unfortunately many designs and implemented hardware look at mass reduction as a pure materials handling reduction in terms of weight per se. It's quite another thing to be concerned with the degree of representativity of the reduced mass fractions.

Also, usually emphasis is on getting a valid analytical result, in the sense that the amount of the analyte in the final sample, a_S, makes do—while TOS emphasizes that only the corresponding estimate of the lot concentration, a_L, carries the information sought. There is a world of difference between these two concentration estimates—the entire

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Fig. 1. Do not focus on directly getting the final analytical volume too early in the sampling process—representative mass reduction does all the work.

1:100 to 1:100.000 mass reduction lies in-between! In the present work we focus on how to reduce the size of any sample (lot, or primary sample) without sacrificing the crucial representativity prior to analysis.

Here we shall not discuss the issues concerning how to do the primary sampling, as this is amply covered in the basic sampling literature [1–14]. Here we are exclusively oriented towards the subsequent mass reduction process(es) involved (principles, methods and hardware: design and maintenance). Even when the extracted primary sample is representative of the lot, it will still be up to the subsequent mass reduction process whether the secondary-, tertiary-, laboratory- and instrumental sample preparation sub-sampling leads to the desired results or not, i.e. whether the mass reduction is ''correct'' or not in the full TOS sense [1–3,14].

We intend to show that making sure that all mass reduction steps are correct allows for a certain indispensable freedom in the sampling process in the sense that one is now free to make the primary sample mass, MS, of any size necessary (due to the heterogeneity of the material, etc.). This means that having to take a large primary sample is no longer a problem. One simply has to reduce this mass before transportation, storage or analysis in order to save time and money. Thus proper mass reduction comes to the fore at all stages in a compound sampling/mass reduction staged process. The principles and procedures examined here are all operational over this entire range, from reducing the primary sample (orders of magnitude span 1 kg–1 ton, or more) all the way down to an analytical mass of the order of grams, micrograms or even smaller.

In slightly more detail: In order for all mass reduction methods or devices to work properly it is critical to respect all the key principles of TOS, primarily that all constituent fragments of the lot must have equal, non-zero, probabilities of ending up in the final sample. This necessitates complete randomness in the selection process of the constituent fragments (units, groups or sub-samples). We here refer to the literature on proper sampling [1–14].

The present paper focuses on 17 current methods and devices commonly used for mass reduction, which have been tested and assessed with regard to a number of characterizing parameters, among which the most prominent are accuracy and reproducibility (precision), constituting the definition of representativity [1,2,15–17]. But in the present comparison study we are in fact interested in the quality of both the average composition estimates resulting from mass reduction operations as well as in the variances of repeated assessments of the performances of the various instruments employed (replicating the entire mass reduction process). Also, other, more practically related parameters such as operating time consumption, user-dependency and device cleaning requirements, etc., are included in the final overall presented below.

This study is complementary to the one by Gerlach et al. [17], who performed a survey of five field-sampling techniques. Gerlach et al. was interested in testing robust, quick and efficient methods for soil splitting in the field (methods included were riffle splitting, paper cone splitting, fractional shoveling, coning and quartering and grab sampling, three of which are also covered here), whereas we are more oriented towards major undertakings associated with industrial and routine laboratory sampling in general. One major difference is that whereas Gerlach et al. only used synthetic samples, we use naturally occurring materials making up 99.90% of all compositions investigated.

2. Material system and analytical procedures

Which material system for comparison purposes would be optimal? Should the material system reflect one dominant situation (necessarily with a relatively smaller range of potential applications fields) or should one strive for as general a material system as possible? What would constitute the latter? This issue is intimately related to the very purpose of mass reduction—here mass splitting is specifically used for the purpose of representative sampling, so the possibility to make generalizations from our survey is of prime importance. Accordingly we have laid down the following criteria for the design of an optimal comparison material system:

(1) The system must reflect both major concentrations, intermediate as well as trace concentrations. For this purpose we have chosen the following levels: 89.9%, 10.0% and 0.1% (1000 ppm).
(2) The material system must be sensitive to flow segregation (indeed also to all other manipulation segregations as far as possible: roll segregation, etc.). This is in order for the system to exhibit a significant degree of segregation as an inherent part of the mass reduction process. We have chosen one component (0.1%) with a very smooth surface (the trace concentration component), one smooth component (10.0%)

and one with slightly softer surface characteristics (89.9%).

(3) It is equally important in the present context that at least some (one, two) of the chosen components also show a significant propensity for "rebounding" when impacting on hard surfaces, as this is an inherent weakness in the design of some mass reduction tools (while being better counteracted by others).

We have stipulated these requirements in order for the comparison system to represent a fair *worst case scenario*; we wanted to test the 17 approaches to be compared exclusively from the point of view of their performance in such a realistic, difficult situation. It is of course trivial to generalize to less adverse situations.

The result was a system of mixed wheat grains, rape seeds and glass spheres, with concentrations 89.9%, 10.0% and 0.1% w/w, respectively. We deliberately chose glass spheres as the trace component, in order to represent, e.g. an impurity (an artifact component), so we did not object to this being an artificial component. We also took great care in designing a material system in which the average grain size, and density, for all three components were not significantly contrasted, in order not to end up in pathological situations (extreme size and/or density agitation segregation). The average grain sizes of wheat, rape seed and glass spheres were: 6.0 (by 3.0 as a "cylinder"), 2.6 and 1.0 mm, respectively. Their average densities were: 0.75, 0.77 and 2.60 g/cm^3. We believe that the chosen system does a good job standing in for a very wide range of industrial and laboratory material systems of aggregate materials and powders with respect to these physical design characteristics. It is admittedly very sensitive for flow segregation, but so much the better when the objective is to test the performance of purported universal mass reduction tools.

Mixing of this lot material prior to all mass reduction experiments (always carefully weighed in completely identical proportions) was carried out by randomly shaking a plastic bucket for 2 min (mechanical shaking and mixing). A lot mass of 2 kg were to be mass reduced to get either 100 or 125 g in the final sample, depending on the nature of the method or device (i.e. dependent upon which split ratios could be obtained with the specific methods). After every pass of mass reduction, the composition of the resulting subsamples was determined, using a screening system consisting of two sieves and a bottom collecting pan, all mounted on a shaking table (Fig. 2), which collected the wheat, rape seed and glass, respectively. The screen sizes were 2.8 and 1.5 mm. We initially performed a set of screening verification experiments; the results showed that the efficiency of separating the three components used was completely satisfactory since the three components were fully separated.

After separating the different fractions of the final reduced samples—as well as the very important fractions of the left-over material (i.e. material rebounded out of the receptacle bins, etc.) were weighed individually by a laboratory analytical weight. Weighing was chosen as "analysis" because of the minimal error associated with this, estimated at 0.01% relative. The masses were used to calculate the analytical result, a_S.

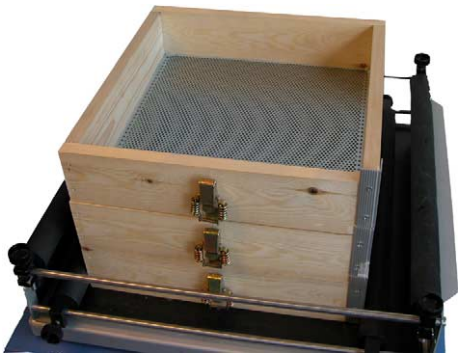

Fig. 2. The screening system.

The same mass reduction/sub-sampling/weighing procedure was repeated 20 times in blocks of 10 by two operators (the two first authors) for all methods and devices investigated. A replication rate of 20 allows for highly trustworthy statistics, which is deemed necessary in order to reach significant conclusions as to reliable, accurate and precise comparability and ranking. Inclusion of two operators in all experiments represents inclusion of inter-operator errors in the overall mass reduction errors estimated in our survey, adding to the validity of a most realistic working situation. If anything, the experimental setup was stacked to reflect a (very) difficult situation indeed.

3. Devices and methods

3.1. Riffle splitting

The most well-founded method for mass reduction is riffle splitting. Riffle splitters can be constructed in several different ways, of which many are in accord with TOS principles and equally many are not. If designed and used correctly it provides a very stable, reliable and inexpensive method for mass reduction with reasonable speed.

3.1.1. Principle

The general principle is that the sample to be divided is introduced to a rectangular area, divided by parallel chutes leading to two separate receptacles. For this device to work properly it must be designed according to a few essential rules. There have to be an equal number of chutes of which every second leads to the two alternate receptacles. The chutes must all have the same size and form; the wall material must be thin in relation to the wall-to-wall dimensions of the chutes themselves. It is also important that no chute can be over-represented when introducing the sample

into the device, for instance by a non-correct design of the sample holders or by a cone-shaped inlet collar in the longitudinal direction. The higher the number of chutes, the better the device splits the sample, both in terms of the splitting bias between the two splits as well as with regard to the variance of repeated splits, as shall be amply demonstrated below. The width of the chutes also has to have a certain minimum width which depends on the particle size, in order to prevent blocking (large particles) or bridging (powders) [18]. An empirical rule-of-thumb stipulates that chutes must be wider than three times the maximum particle size or two times this plus 5 mm, since even extremely small particles should not be split using smaller chute width than 5 mm. The general literature on TOS has exhaustive analysis and discussions of correct design principles of riffle splitters, which must be consulted before acquisition of any riffle splitter [1,2].

3.1.2. Use of riffle splitters

It is, however, not enough to have access to a correctly designed riffle splitter. In order to obtain a representative mass reduction, the device also has to be used—and indeed cleaned and maintained—correctly. There are a few simple rules that must be followed, which may be summarized as follows:

1. The sample must be spread out equally over the whole length of the feeding tray.
2. The feeding tray must have exactly the same width as the rectangular receiving region of splitter; there is thus no need for inclined inlet collars, etc., in the longitudinal direction.
3. The sample must be fed perpendicularly to the longitudinal axis along the device; the sample must be fed precisely on to the center axis.
4. No particles can be allowed to bounce out of the receiving trays or the splitter.
5. The split sample (or the portion to be split further) must be chosen at random.

If these rules are obeyed, any split portion should (in theory) not be systematically biased by the splitter. Fig. 3 shows some of the errors than can result from incorrect design and use of riffle splitters. To understand the importance of the design it is important to remember that even though the sample is evenly spread over the width of the feeding tray, it cannot in practice become homogeneous and this will lead to (minor) differences between the feed for the individual chutes.

3.1.3. Device description

In the present work six different riffle splitters of the basic design described above were tested. During the experimental runs several optimizations on existing devices and the design of a new device took place. This is described in further detail in a later section. The splitters used are named according to design and for easier distinction as follows:

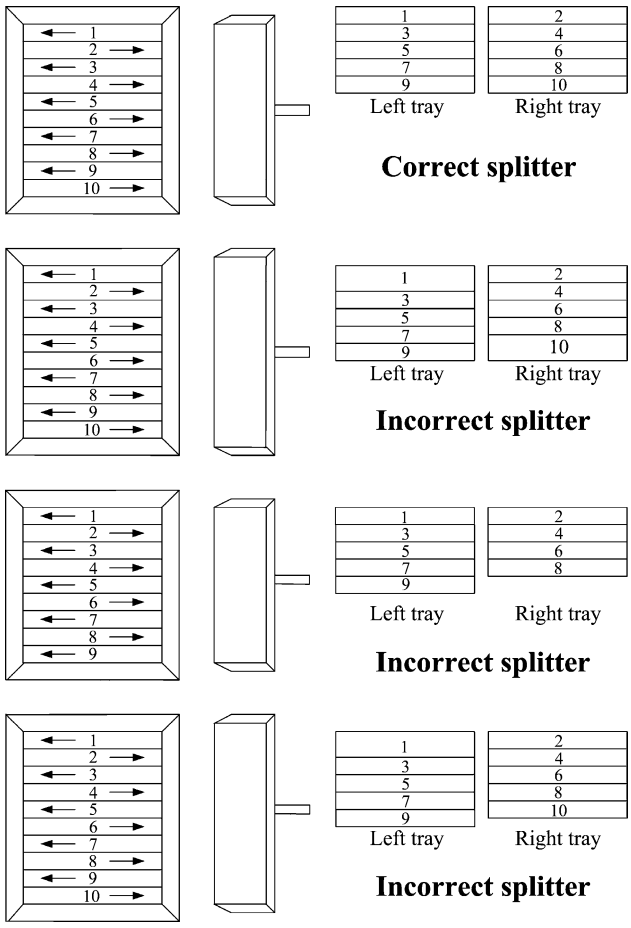

Fig. 3. Schematic illustration of the critical importance of correct riffle splitter design.

- The animal feed splitter
- The seed splitter
- RK 10 chutes/20 mm width splitter
- RK 10 chutes/30 mm width splitter
- RK 18 chutes/16 mm width splitter
- RK 34 chutes/10 mm width splitter

The latter four are manufactured by the same company and three of these are designed in an exactly identical fashion, only scaled-up. The 34 chute splitter differs, since it represents a completely new design resulting from the present work. In the following sections the individual splitters are further described.

3.1.4. The animal feed splitter

This divider (Fig. 4) has 10 chutes and is used by the Danish Ministry of Agriculture's department of animal feed testing. The chutes are 27 mm wide. The design has several apparent errors, but also some advantages. The device has three identical trays, two used for receiving and one for

Fig. 4. The animal feed splitter.

Fig. 6. The seed splitter.

feeding. The trays can be switched freely, making handling easier. The greatest advantage is that it can be taken apart for easy cleaning.

One of the obvious errors is found in the design of the trays, since these are narrower than the top of the divider and the section for the receiving trays. This makes the introduction of the sample to the divider and the reception of the sub-samples unavoidable sources of bias. If the reception trays are placed wrongly, which easily happens, some of the material is lost completely since it does not even hit the reception trays at all. Fig. 5 shows a close-up of the side of the divider when one of the receiving trays is placed wrongly. It is observed that almost half the material hitting the uttermost chute will be lost.

Another error is the open design of the top of the splitter, where many particles (especially rape seed) are observed to bounce out of the device during operation. The Feed Splitter is greatly dependent on the user because of the pouring mechanism. This cannot be avoided in the current design.

3.1.5. The seed splitter

This device (Fig. 6) is used by the Danish Ministry of Agriculture's department of seed testing and has 20 chutes of width 10 mm. The design again has errors, but also some good features.

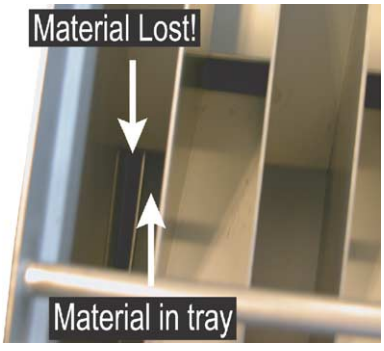

Fig. 5. Unwanted design error for the animal feed riffle splitter. If the tray is placed wrongly, as is very easy, almost half the material hitting the most peripheral chutes is lost.

The errors consist of the open design of the section between the feeding tray and the chutes. Much material is lost in this region due to particles bouncing out of the splitter. The advantages are found in the feeding mechanism, which makes handling easy and also minimizes the possibility of operator introduced errors. This splitter can also be taken apart for easy cleaning. The relatively high number of chutes (20) makes the splitter more reliable than the previous one. A nice detail is that the leading edges of the blades between the chutes are sharpened to minimize particle bouncing. The feeding tray is nicely aligned with the sides of the splitter and no error is thus induced from this.

3.1.6. The RK splitters

Three of the four splitters from "Rationel Kornservice A/S" (RK) are designed from the same basic principles. The only features changing are the number of chutes, the width of these chutes and the resulting overall dimensions of the devices. The splitters consist of two separate reception trays, a splitter and a swinging, mounted feeding tray (which can be easily dismounted however if need arises). These splitters are delivered with extra plates for insertion over the chutes to minimize sample loss due to bouncing. The reason that these insertion plates are not permanently installed is only that the splitters are also used for grass that has a tendency to clog up the device if this inner clearing is too narrow. These plates were installed on all the splitters used in the current experiments, except the RK 10 chute (20 mm) and RK 34 chute (10 mm) splitters.

There are errors in this design too. The first relate to the fact that the feeding trays are slightly narrower than the top of the splitters. This, however, is possibly only of marginal importance, since the error is the same in both distal ends, and thus both reservoirs are underrepresented from the outmost chutes by approximately the same amount. The splitter is not easily cleaned since it cannot be taken apart. The advantage on the other hand is equally obvious since a

Fig. 7. The 10 chutes/20 mm width splitter (left) and the 18 chutes/16 mm width splitter in action (right). Notice the closed design resulting in minimal material loss.

minimum of particles are lost during use because of the closed design; another advantage would be that the sample is presented to the splitter in exactly the same way every time, because of the fixed feeding tray. Fig. 7 shows the principle design and use of these RK splitters.

The last of the four RK splitters is a result of an ongoing collaboration between ACACSRG and Rationel Kornservice A/S to build a better splitter. The new design has several improvements, mainly in the increased number of chutes (34) and the optimized feeding tray. In the next section this device is described in detail.

3.1.7. RK 34—device optimization

The new design differs from the old mainly in the feeding device and the number of chutes. Designing the new feeding mechanism started by checking out the idea of using the same principle of a mounted feeding tray as the seed splitter. This turned out to be a very constructive idea and the design process continued on this basis. The solution sought had to eliminate the step of pouring material from one of the previous receiving trays into the feeding mechanism, since this introduced rolling and trajectory segregation as well as impact effects. When spreading out the material as evenly as possible in the feeding trays, these effects lead to bias of the results. The final solution was simply to furnish the feeding mechanism with a slot for directly inserting one of the previous used receiving trays (Fig. 8).

When tipped, the sample is poured into the conically inclined interior of the feeding device. This minimizes the effect of segregation drastically. The conical delivery funnel only opens when the tray is tipped all the way and gently brought in contact with the chute area in the splitter due to a small activation/stopper pin. Also, the width of the opening can be regulated by this controlling pin (Fig. 9).

Several lengths of the feeding funnel were tested out, to see if a shorter delivery path along which the particles can segregate would increase splitter precision of repeated operations. This was not the case and therefore the final design was kept to minimize size and weight configuration for both economical and practical reasons. The resulting

Fig. 8. The final RK 34 design, using an insertion slot for a third tray in the feeding device.

splitter looks exactly as in Fig. 8, but with a shorter feeding funnel. As in the previous design there is indeed in principle introduced a very small error since the feeding tray is slightly shorter than the splitter length by the width of two individual chute walls; this would appear almost totally negligible however, since in the longitudinal direction this foreshortening amounts to 1.6 mm/359.6 mm, or 0.4% only. Since we were in fact unable to demonstrate any effect of this error, the design was consequently kept as is. The results of the present overall survey were not known to us at the time when we decided to stop the development process of the present apparatus. Only later it was learned that this prototype RK 34 riffle splitter indeed outperformed all other riffle splitters in the present study, so this error truly must be exceedingly small. For more homogeneous systems the old (long) feeding trays can still be mounted, since these fit into the same socket as the new one. This also opens the possibility of changing to the new feeding mechanism on existing dividers, etc. RK has since the ending of the present work declared that all future splitters (all sizes and models) will be built according to this new design.

Fig. 9. The feeding tray opens "automatically" when tipped and gently pressed against the chutes. This takes place exactly at the center longitudinal axis. The small pin is inserted for controlling the width of the opening.

3.1.8. Experimental run

The riffle splitter experiments were carried out in identical fashions for all methods. The 2 kg were divided four times to get approximately 125 g in the final sample. After each split the tray to be further divided, or the final sample, was selected at random. The practical procedures for the individual splitters were necessarily a little problem-dependent, but all efforts were made to follow the rules of correct use. The material was poured into the feeding tray and spread evenly over the entire width with the greatest care in order to allow all methods to compete evenly and fairly. The material was then fed along the center axis of the chutes with a steady flow in order to minimize bouncing, especially of rape seed. All RK riffle splitters tested were fast and easy to use. The three older designed splitters can all be somewhat user-dependent, since pouring the sample into the feeding tray can vary. By using the improved design this user-dependency is minimized or fully eliminated.

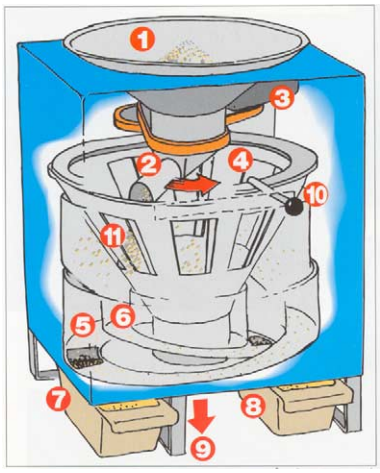

Fig. 10. Vario Divider. 1: hopper, 2: revolving feeder, 3: motor, 4: chute closer, 5 and 6: sample outlets, 7 and 8: samples, 9: left-over material, 10: chute opening handle and, 11: chute [19].

3.2. Revolving splitters

Revolving splitters are based on the same principle as riffle splitters. The revolving feeder distributes the sample material equally (in time) over a number of radial chutes, assuming constant rotational speed. These devices are very easy to use, since one only needs to pour the material to be divided into a hopper, thereby getting one or several reduced splits. They also require very little preparation and clean-up and split the sample very fast. The latter of course depends on the rotating speed as well as the influx velocity of the material through the feeding funnel. The essential principle here is that every second radial chute contributes to one of the two alternative collecting reservoirs.

Since a larger number of sub-samples in this context give more representative samples it can be desired to increase the total number of revolutions. This can be done by using a smaller outlet size of the hopper. Also, size, slope and rotation speed of the inlet tube can be altered to change the outlet speed.

3.2.1. Vario Divider

With a revolving variable sample divider named "Vario Divider" (Fig. 10), it is possible to get a mass reduction ratio as small as 1:100, depending on the nature (mainly particle size) of the material to be split. Some models include the possibility of getting several final samples.

The lot material is poured into a hopper (1) from where it is led to a revolving feeder (2). From here the material is led either to a chute opening (11) or to the bottom as left-over material (9). The divider shown gives two equivalent samples (7 and 8).

When using the model type 1G/1−4 on the current sample composition, it is only possible to get a sampling ratio of 1:9 because of the particle sizes involved. To obtain a sample of 100 g it is necessary to realize a sampling ratio of 1:20. The sample therefore had to be divided in at least two steps, which allows for a realistic testing of the Vario dividing principle.

Two different multiple-step settings were tested: sampling ratios of 1:4 + 1:5 and 1:2 + 1:2 + 1:5. Splitting the sample in three steps is of course more time demanding; however, we found this to be only minimal in practical terms. In general, using more steps results in a larger sampling error since every single step is error generating. Again, this is setting the comparison study with the most stringent demands on the performance for this device type. Only a marginal sample loss was observed. This was exclusively caused by rebounding of the rape seeds from the sample boxes. Therefore almost no maintenance or clean-up was necessary, and the method must be classified as very easy and fast to use. Since the user only has to pour the sample into a hopper, neither any user-dependency is observed.

There has been a certain theoretical discussion regarding the possibilities of such devices to deliver correct (representative) split samples, mainly related to the variable portion of left-over material—we here refrain from entering into this discussion, but are the more happy to include examples of these Vario Dividers into the set of devices to be compared and ranked. We will let the empirical performance of these revolving riffle splitters speak for itself.

3.2.2. 32-Divider (fixed ratio)

With the 32-Divider (fixed ratio), Fig. 11, it is possible to get the lot material divided into 32 supposedly equal subsamples (so the design objective claims).

The principle used for this divider is identical to that for the Vario Divider, but without any variability whatsoever. The whole cross-section area is divided into 32 fixed chutes, so that the lot material is split completely, and there is no left-over material at all. The constant rotation of the revolving feeder causes the lot material to be equally divided amongst the 32 chutes giving 32 sub-samples.

Fig. 11. The 32-Divider.

Two of the 32 sub-samples were joined into one composite sample of approximately 125 g; these two sub-samples were always selected at random from the 32 candidates. The tested divider was fairly easy to use, but required the user to attach plastic bags to each of the 32 tubes. This did of course affect the preparation time for each split, but since this is only a practical, and easily solvable problem, it should not be taken into serious account. Besides this minor attachment work it was regarded as a fast and easy mass divider to use, with no maintenance. If the sub-samples are extracted at random, neither systematic differences nor user-dependencies are expected to influence the splitting results.

3.3. Shoveling methods

3.3.1. Spoon Method

The Spoon Method can be used to achieve relatively low sampling ratios. This method is somewhat related to the principles behind bed blending, but weakly at best [1,2,20]. The lot material is spread out in an even layer: the lot is poured out on a flat surface as a thin string in an s-shape movement in one direction. This is subsequently repeated in the traverse (90° rotated) direction, then this procedure is repeated again and so forth until all of the lot is poured into the tray, as shown in Fig. 12.

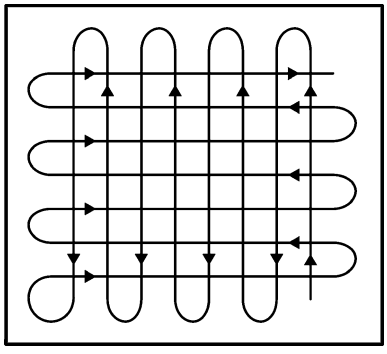

Fig. 12. The pattern used for spreading the lot material in the tray.

Fig. 13. Extraction of sample using the spoon method.

After the laying out step, the spoon method simply consists in extracting a "sample" by using a spatula and a small spoon ensuring that the bottom of the tray is reached (Fig. 13). Several sub-samples, increments, are extracted and joined as the final sample, reducing the effect of the grouping factor [1,2]:

$$\gamma = \frac{N_F - N_G}{N_G - 1}$$

where N_F is the number of fragments in the lot and N_G is the number of groups, or increments, in the lot.

It is important that the final sample is composed of as many increments as possible. To make sure that uncompromised increments can in fact be extracted, and thereby reducing any incorrect sampling errors (ISE), the lot material has to be spread out in a layer of a certain thickness. The extracted sub-samples have to be extracted completely at random from the whole lot to get a probabilistic sample. The method is generally time consuming, tedious, and greatly user-dependent. By following the guidelines mentioned above the user-dependency will be somewhat reduced.

In practice, the final sample in the present experiments was composed of five increments (Fig. 14), which resulted in an average mass of 115 g. Some sample loss always occurred due to problems (selective losses) during increment transfer to the sample box.

3.3.2. Alternate shoveling

The alternate shoveling method can be used to split a sample into two samples with almost equal weight, hope-

Fig. 14. Box after random sample extraction by the "spoon method".

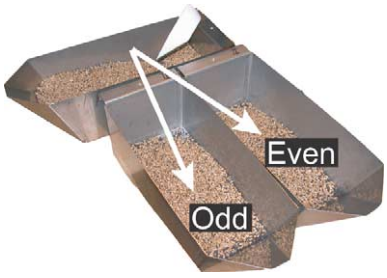

Fig. 15. Alternate shoveling.

fully also of almost equal composition. TOS has made thorough analysis of this general approach [1–3], and there are many pitfalls which are almost universally unavoidable in practice. The equality of the final samples obtained by this method will also be highly dependent on the nature of the material sampled.

The method is based on the principle that all extracted shovelfuls from the original sample are deposited sequentially in two alternative heaps as illustrated in Fig. 15.

It is important that all extracted shovelfuls are selected at random from the initial lot and that all increments have the same (approximate) size. Each heap should consist of an equal number of shovelfuls. One heap should only consist of all even-numbered samples while the other should only consist of all odd-numbered samples. By ensuring that all shovelfuls are carefully selected at random, the condition of sampling equity is preserved thereby minimizing the risk of a systematic bias to some degree.

Four full splits were necessary to obtain a final sample on approximately 125 g in our experimental runs. Some sample loss was observed due to the practical handling of shovelfuls. Different samplers (operators) will definitely have unequal impact on the quality of the final reduced samples, since the shoveling can vary greatly from user to user.

3.3.3. Fractional shoveling

With true fractional shoveling it is possible to divide the lot material into N sub-samples instead of only two. Shovelfuls are extracted from the lot material and deposited into N distinct heaps. In Fig. 16, true fractional shoveling with $N=5$ is shown.

Fig. 16. Fractional shoveling with $N=5$.

Fig. 17. Grab sampling.

The shovelfuls should again all be extracted at random from the lot material and should be (approximately) equal in size. Each heap should consist of an equal number of shovelfuls. All extracted shovelfuls should be alternated from heap one to heap N.

To reduce the initial 2 kg into approximately 100 g the sample was first split into five heaps. From these five heaps one was chosen randomly and further divided into four samples at approximately 100 g each. This method can also be slow, tedious and user-dependent.

3.3.4. Grab sampling

Grab sampling is the easy choice for extracting a "sample" and is (unfortunately) very often the preferred choice in practical sampling situations. One sample only is extracted to represent the whole lot. Grab sampling is the archetype sampling error at work. The focus is exclusively on getting the final sample mass directly in one go! However, a grab sample may also result from joining several increments (sub-samples), to produce a composite sample, which in general should result in a more representative sample. Grab samples are typically taken by a

Fig. 18. Boerner Divider.

Fig. 19. Boerner Divider at work.

scoop or a shovel, depending on the size of the original lot, etc.

To obtain a truly representative sample all virtual units making up the lot per force must have the same probability of being selected. With grab sampling this manifestly neither can be, nor hardly ever is, the case. The singular sample to be extracted by grab sampling is of course also often taken from an(y) easily accessible part of a lot, i.e. the top. Grab sampling is therefore often classified as deterministic sampling.

Extracting a sample from the bottom of the lot in Fig. 17 was difficult as in most cases (extraction shown in the figure). The sample domain was divided into six virtual areas. Each grab sample was extracted at random from one of these six areas thereby counteracting unwanted systematic bias. It was attempted to extract the sample from the whole virtual area by pushing the scoop to the bottom and withdrawing it slowly upwards.

If such ‘‘samples’’ are erroneously accepted, grab sampling is a fast and easy choice. If the final sample is to be a probabilistic sample it can be time consuming, very often difficult or downright impossible to get correctly sampled increments. Lastly grab sampling is always user-dependent. Following TOS, grab sampling is supposed to perform the worst of all alternative mass reduction approaches. It was therefore a natural must for the present comparison purpose, if nothing else as a (bad) benchmark.

3.4. Other methods

3.4.1. Boerner Divider

Using the Boerner Divider (Figs. 18 and 19) it is possible to divide a sample into two app. equal half-splits. The halved samples can iteratively be divided again, etc., until a satisfactory sample size has been reached.

The initial sample is poured into a hopper. When the hopper’s bottom-shutter is opened the sample is directed down onto the top of a cone. Since the sample is flowing downwards by gravity it should be spread out evenly in all azimuth directions. At the bottom of the cone the material is led through a number of alternative chutes which are so connected so as to lead the alternate part-streams into the resulting two half-sample splits.

The Boerner Divider is very easy and very fast to use. Set-up and maintenance is straight forward, but some sample loss can occur during use, depending greatly on the nature of the material to be split. Especially bouncing materials such as rape seed and glass pellets can be a problem. To avoid any systematic differences from one side of the splitter (receiving tray) to the other it is also here important to choose one of the two receptacle trays randomly.

The initial 2 kg material lots in the present study had to be divided four times to get a sample size of approximately 125 g. Some loss of material was observed primarily due to the design of the receptacle tray combined with the nature of the glass and rape seed used.

Two different Boerner Dividers were tested; one will be referred to as calibrated, the other as non-calibrated, mean-

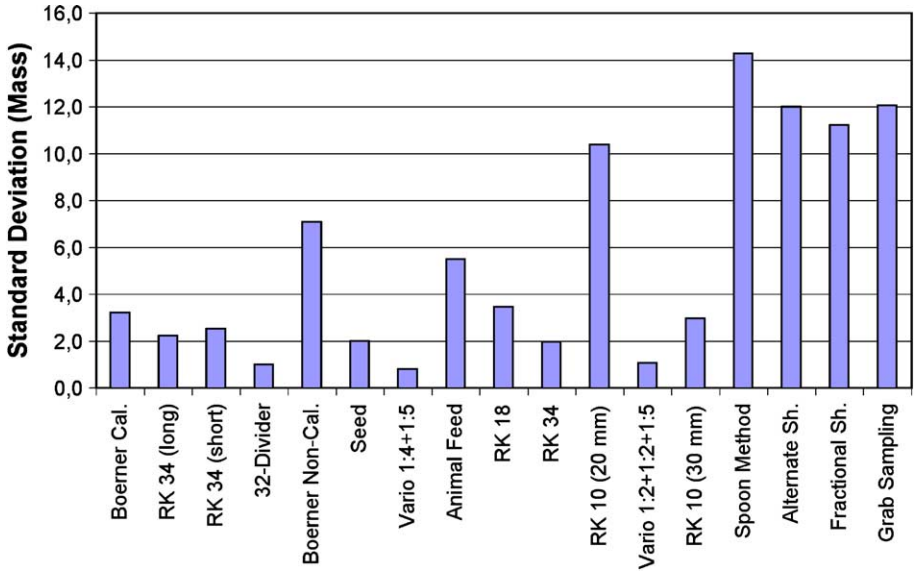

Fig. 20. Standard deviation of the final sample mass.

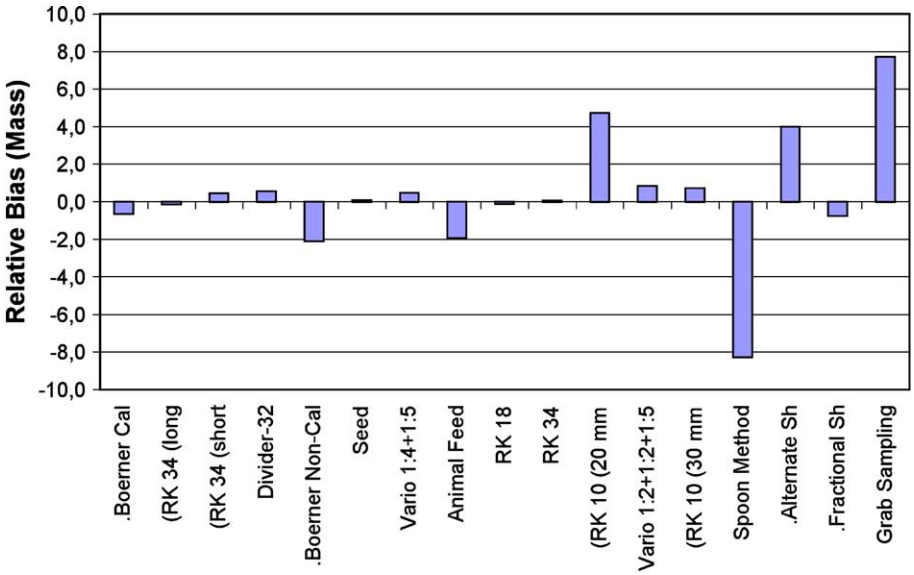

Fig. 21. Relative bias of the final sample mass.

ing that the hopper was slightly off-centered and that the receptacle bins where open. For the non-calibrated divider this could lead to over-representation of some chutes and a fair amount of lost material. Figs. 18 and 19 show the non-calibrated divider.

4. Results and discussion

4.1. Characterizing parameters

In order to evaluate and compare the above methods a system is developed for characterization by a selected set of mass reduction quality parameters. These parameters are described shortly below.

4.1.1. Mean and bias (concentration)

After performing the splits or the individual mass reductions, the results are characterized by the arithmetic average concentration for all three materials after the universal 20 repetitions. This parameter is very important in practice since it characterizes the method's ability to perform splits and leave the sample with the same composition as the lot material.

The mean is a measure of the accuracy of the method when assessed against the true x_{Lot} (a_{L}). The bias is simply

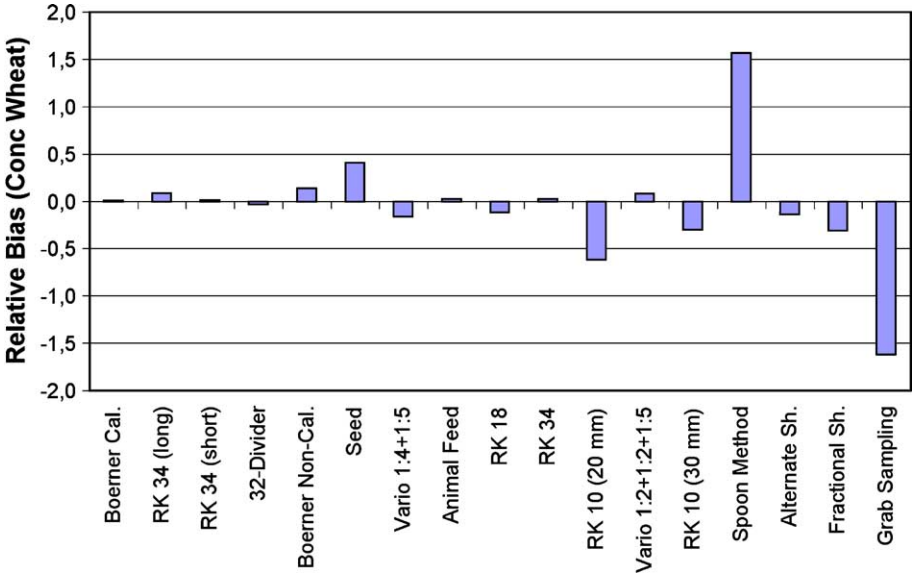

Fig. 22. Relative bias for the concentration of wheat.

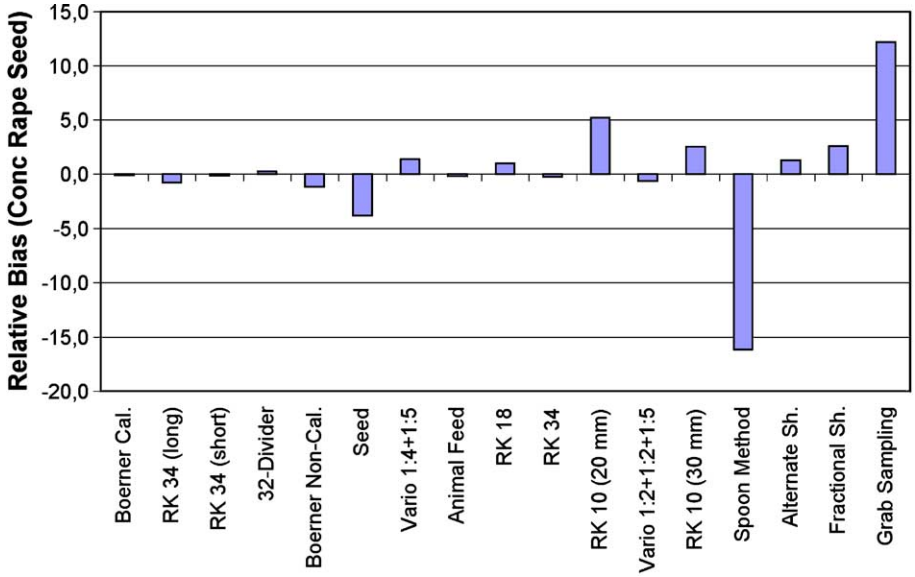

Fig. 23. Relative bias for the concentration of rape seed.

calculated as the average concentration minus the true (known) concentration of the lot, x_{Lot} (or a_{L}). The bias is also presented as a relative value showing the percent wise deviation from 89.9, 10.0 or 0.1% w/w. The relative bias is calculated as:

$$\text{Relative bias} = 100 - \frac{100 \cdot x_{\mathrm{Lot}}}{\bar{x}}$$

4.1.2. Standard deviation (concentration)

In order to characterize the dispersion of the results around the mean, the standard deviations are calculated. The formula used is based on sample statistics (i.e. a statistical sample selected from a large population), since 20 results is not (statistically speaking) really a large number of replicates. The square of the standard deviation, the variance, represents a measure of the reproducibility of the method (precision in statistical terms).

4.1.3. Mean and bias (mass)

The ability to extract the wanted mass of material for the sample is characterized by the mean of the masses. This is an important factor in industrial, laboratory as well as field sampling, where samples often have to be of more or less constant mass.

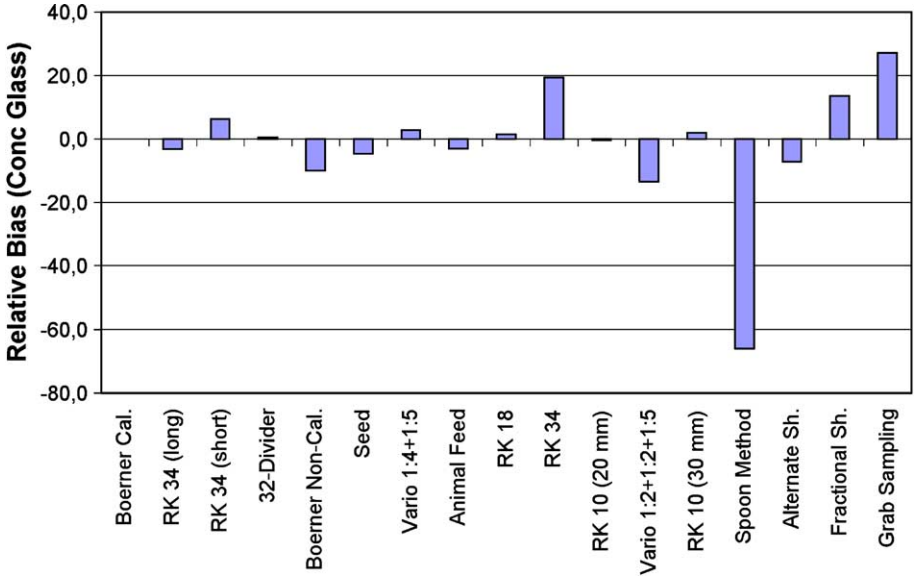

Fig. 24. Relative bias for the concentration of glass.

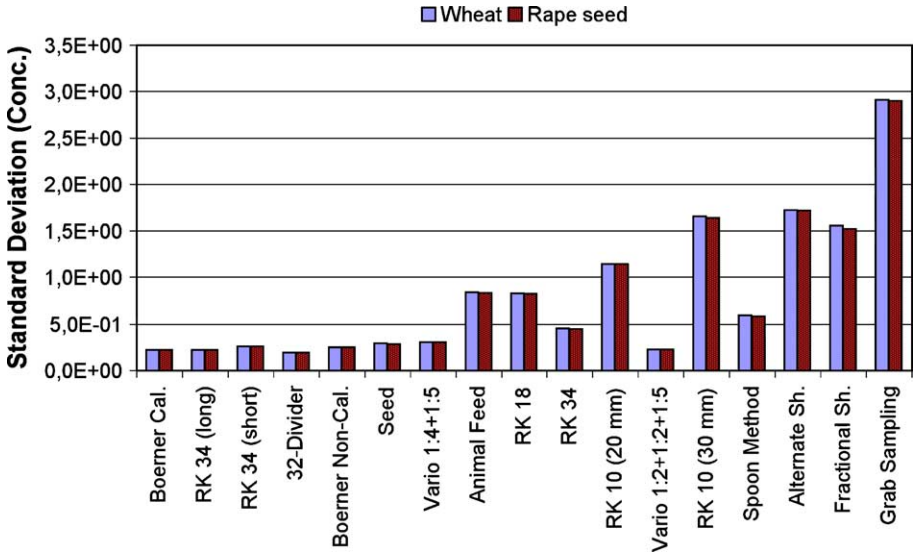

Fig. 25. Standard deviation for the concentration of wheat and rape seed.

4.1.4. Standard deviation (mass)

Again, the dispersion of the individual runs around the mean is presented as the standard deviation.

4.1.5. Loss of material

In order to characterize the method it is also interesting to find out to what degree it causes loss of material during mass reduction. This parameter is also related to the recovered mass of the three fractions in the sample as well as to the residue (material adhering to walls or other surfaces in the apparatus after completed splitting); the residue is estimated as the mass of cleaned-out material. Loss is simply calculated as the initial mass (2 kg) minus the sum of these two recovered weights.

4.1.6. Representativeness

Representativeness is authoritatively defined by Gy [1] and is the only statistic term that includes both accuracy and reproducibility (precision). Many authors, standards and norm writers use the term representative only very loosely and very often without a proper mathematical definition. Normally this characteristic is used for setting a lower limit, with which a representativity statistic for a particular split has to comply in order to be acceptable. In this work

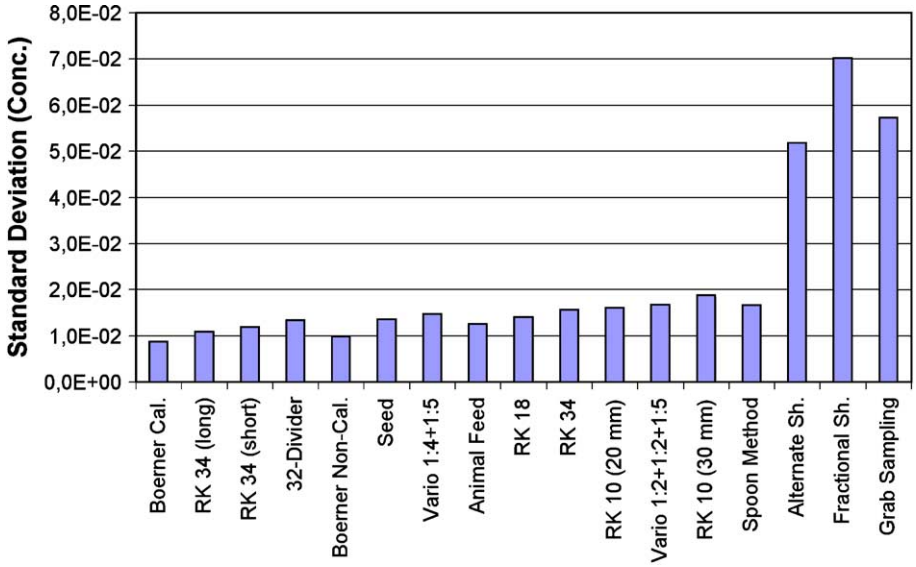

Fig. 26. Standard deviation on the concentration of glass.

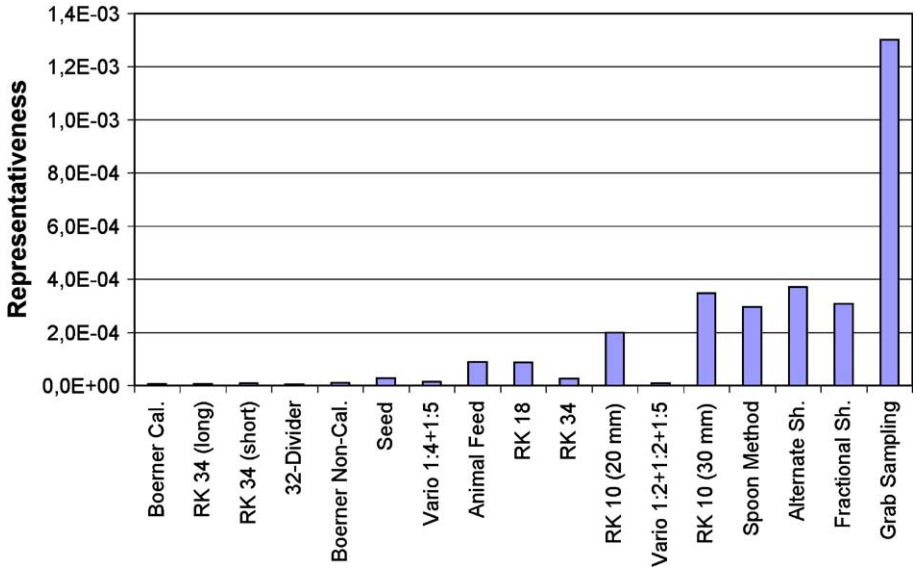

Fig. 27. Representativeness of wheat.

however we use it strictly for comparative purposes. The representativeness is defined as the mean square of the selection error, SE, i.e. the sum of the squares of the mean and standard deviation of the selection error [2]:

$$r^2(\mathrm{SE}) = m^2(\mathrm{SE}) + s^2(\mathrm{SE})$$

where the selection error in turn is defined as:

$$\mathrm{SE} = \frac{a_\mathrm{S} - a_\mathrm{L}}{a_\mathrm{L}}$$

Here a_S is the grade of the critical component in the sample and a_L is the grade of the critical component in the lot material. The latter is known without any uncertainty in our runs, since we carefully prepared the same initial lot composition for each experiment.

4.1.7. Processing time

In order for the method to be attractive, for instance for handling a high throughput of samples (commercial laboratories, government and other regulating bodies, etc.), processing time has to be relatively low. The parameter presented here is simply the average time duration, in seconds, from the beginning of the split until the final sample is at hand.

4.1.8. User-dependency

This characteristic is meant to divide the methods in two types; the first of which is where the person performing the

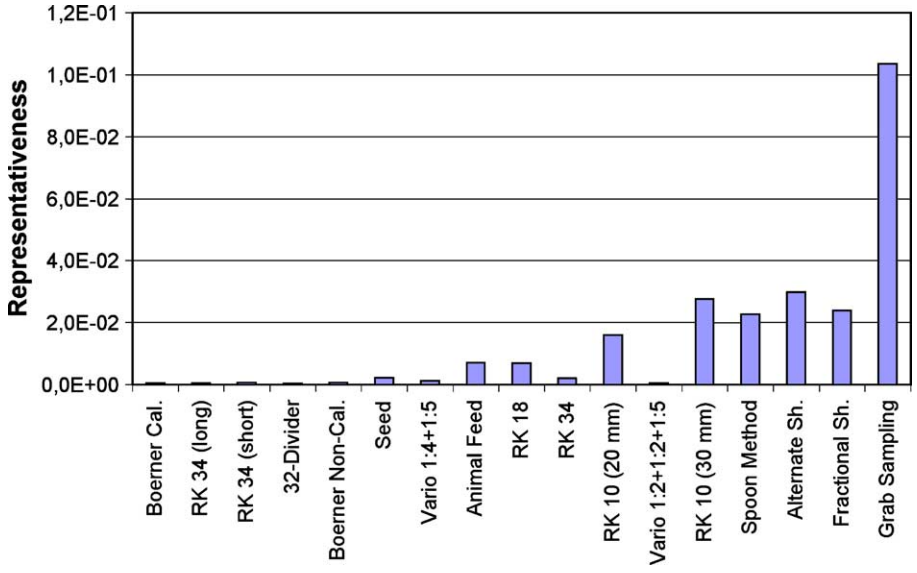

Fig. 28. Representativeness of rape seed.

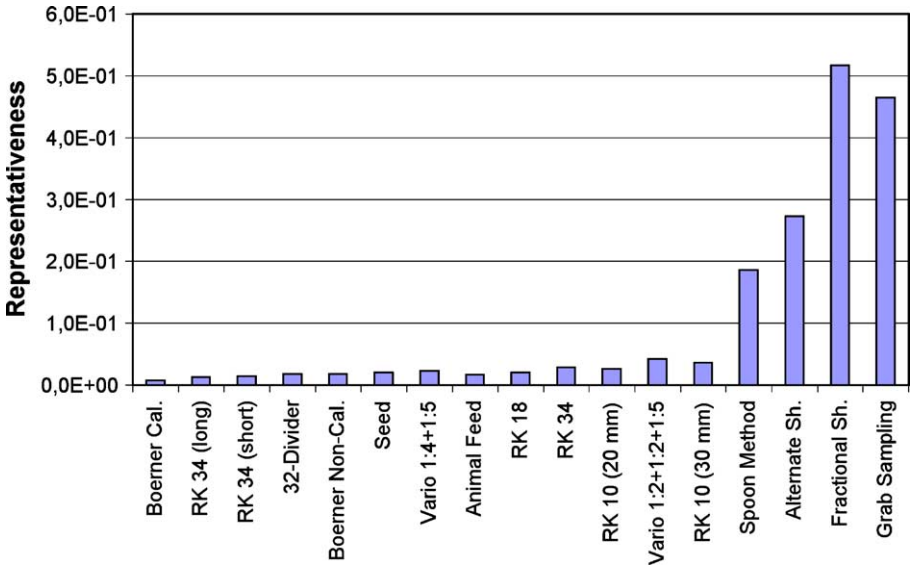

Fig. 29. Representativeness of glass.

sampling has a large influence on the result. The second section is where this influence is minimized if not totally negligible.

4.1.9. Cleaning

Some devices are easily cleaned and others are more difficult to dissemble, etc. This is an overall assessment performed by the authors in every case. It is divided into three categories: easy, intermediate and hard.

4.1.10. Initialization

Another parameter that might be of comparative importance would be the time consumption and workload needed in initializing a mass reduction operation. For instance when the same instrument has to be used for different material systems, or for performing different split ratios, initialization time might be important. Again, this is an assessment performed by the authors and it is divided in three groups: quick, intermediate and long.

4.2. Comparison of mass reduction methods

There are two major fields of interest when characterizing a mass reduction method: the mass of the final sample and the composition resulting from analysis of this mass. The ability of the devices/methods to find the correct (target) mass is summarized in Figs. 20 and 21. We notice that the shoveling methods show a particularly bad accuracy

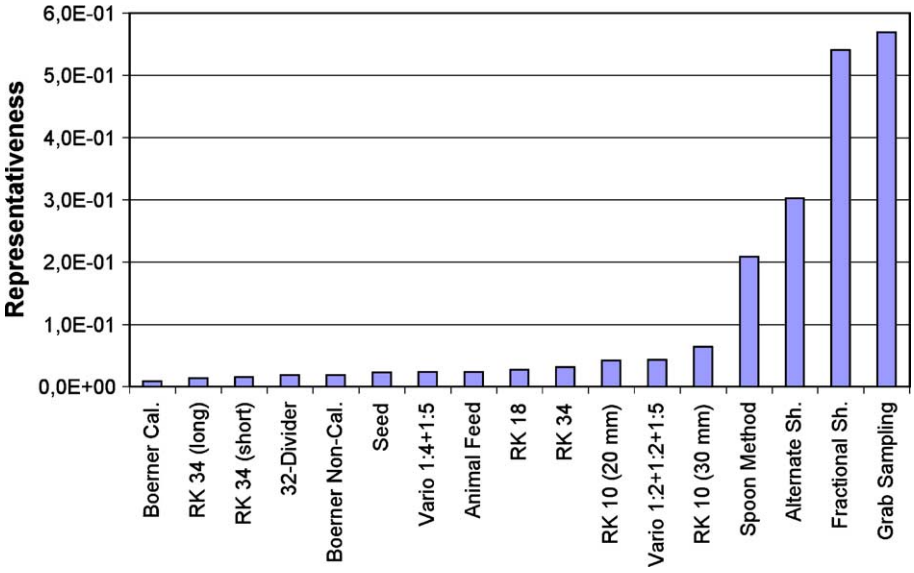

Fig. 30. Sum of representativeness (pooled for wheat, rape seed and glass).

and precision. Only fractional shoveling seems to have a relatively good precision. This method is comparable to alternate shoveling, but differs in having only two mass reduction steps instead of four, which possibly can explain the better precision. The rotational dividers and some of the riffle splitters seem to have good accuracy and precision throughout in finding the target mass. The RK 10 chute (20 mm splitter), however, differ significantly from the rest of the riffle splitters (in an adverse sense). The only possible reason for this must be the missing insertion plates on this splitter.

With regard to the composition of the final sample, the methods are evaluated by the standard deviation, relative bias and the representativeness.

In Figs. 22–24, the methods deviating most from the rest clearly are the spoon method and grab sampling. This is understandable since these two methods are both shoveling methods, and thus expected to be less precise. In general, all shoveling methods and some of the riffle splitters are characterized by bad precision, while all revolving dividers show good precision. The reason for this good precision is the large number of rotations involved, and hence the large number of effective chutes involved in the mass reduction. When the sample takes about 1 min to pass through these devices, the number of revolutions per minute is 40 and the number of openings eight, the effective number of chutes is actually 320 for the Vario Dividers and an impressive 1280 for the 32-divider (which has 32 openings).

The standard deviation is an expression of the precision of a particular method. Fig. 25 shows the standard deviation of wheat and rape seed, which both are present in rather large concentrations in the material (89.9 and 10.0% w/w). All the methods with a large number of chutes or openings have a low standard deviation on both wheat and rape seed. The shoveling methods, again, stand out as terribly imprecise methods, even though the spoon method would appear just within the window for this parameter alone. This is possibly due to the bed blending like preparation of the lot material and the extraction method, which for the experienced operator ensures nicely delimited increments (sub-samples).

Glass is present in very low concentration (0.1% w/w) in the material, and it is expected that the reproducibility (precision) for this material is substantially worse than the components present in larger concentrations. The absolute values in Fig. 26 are not directly comparable with those in Fig. 25, since the standard deviation is a relative value. It is noted that the precision of all the methods is more or less equal for the trace element level, except for the shoveling methods. The spoon method again seems to have an acceptable performance, but the rest of the shoveling methods are distinctly bad, very likely due to the extraction method.

The overall TOS-measure representativeness takes into account both accuracy and precision, and will thus express the overall performance of a method. It is seen in Figs. 27–30 that the methods with the lowest number of chutes or openings and the shoveling methods indeed are worst. This is in accordance with the previous conclusions.

The representativeness of glass has a dominating influence on the pooled sum, since these values are much larger than the values for wheat and rape seed. However, the sum is the best measure for the overall performance of the methods, since it includes constituents present in both high and low concentrations. It is observed from Fig. 30 that the calibrated Boerner Divider has the best overall performance. There is, however, no large difference to be found between

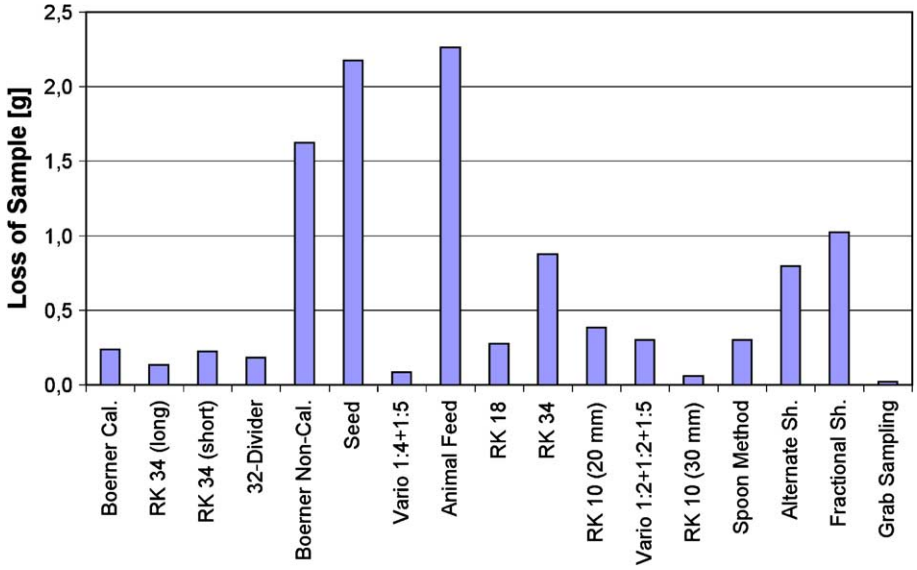

Fig. 31. Total loss of material.

the 10 best of the methods, meaning that all these methods in principle are suitable for mass reduction with regard to representativeness.

The total loss of material during mass reduction is seen in Fig. 31. The loss is high for all splitters with open designs, and for all the shoveling methods with several steps involved. Especially the seed splitter, the animal feed splitter and the non-calibrated Boerner Divider stand out as spilling large amounts of material (especially rape seed is seen to bounce out).

In Table 1, the compound characteristics for the investigated methods are summarized. The different values are here weighed equally and it is left for the reader to apply differential weighing to fit his or her own customs or needs. If for instance operating time is of greater importance than cleaning, or if it is absolutely crucial to have the correct mass in the final sample, these parameters can be weighed on an individual basis.

The resulting sum-scores divide the methods/devices into three groups:

Very Good (sum = 5)
- Boerner Divider (Cal)
- RK 34 chutes of 10 mm short
- RK 34 chutes of 10 mm long
- 32-Divider
- Vario Divider 1:4 + 1:5

Acceptable (sum = 4, 3 or 2) but only under certain, problem-related circumstances:
- Boerner Divider (Non-cal.)
- Seed Splitter
- Rk 18 chutes of 16 mm
- RK 34 chutes of 10 mm normal
- Vario Divider 1:2 + 1:2 + 1:5
- RK 10 chutes of 30 mm

Poor (sum less than 2) not recommended under any circumstances
- Animal feed splitter
- RK 10 chutes of 20 mm
- Spoon method
- Alternate shoveling
- Fractional shoveling
- Grab sampling

The newly developed riffle splitters, the calibrated Boerner Divider, the 32-divider and the Vario Divider outperform all other methods—even though they all can be difficult to clean. The riffle splitters are in general rather slow to use, but this is a relative factor, since the slowest method overall—alternate shoveling—uses only approximately 200 s to reduce the mass by a factor of 20. The devices in the best group are all really good at finding the correct target concentration and mass of a final sample.

This is also the overall conclusion for the intermediate group. Most of these methods have a significant loss however and/or are also rather slow to use.

Table 1
Summary of the characteristics for the investigated methods

	Boerner Divider (Cal)	RK 34 long	RK 34 short	32-Dvider	Boerner Divider	Seed splitter	Vario 1:4 + 1:5	Animal feed splitter	RK 18 chutes og 16 mm	RK 34 Normal	RK 10 chutes of 20 mm	Vario 1:2 + 1:2 + 1:5	RK 10 chutes of 30 mm	Spoon method	Alternate shoveling	Fractional shoveling	Grab sampling
Composition	+	+	+	+	+	+	+	+	+	+	0	0	0	−	−	−	−
Mass	+	+	+	+	0	+	+	0	+	+	−	+	+	−	−	−	−
Loss	+	+	+	+	−	−	+	−	+	0	+	+	+	+	0	0	+
Cleaning	0	0	0	−	0	+	−	+	0	0	0	−	0	0	+	+	+
Initialization	+	+	+	+	+	+	+	+	+	+	+	+	+	0	+	0	+
User-dependency	+	+	+	+	+	+	+	0	0	+	0	+	0	−	−	−	−
Time	+	0	0	+	0	0	+	−	0	0	−	+	0	−	−	−	+
Score	5	5	5	5	2	4	5	1	4	4	0	4	3	−3	−2	−3	1

Table 2
Parameters used to estimate FSE

	M_L [g]	a_L	δ_A [g/cm^3]	c [g/cm^3]	β	f	g	d [cm]
Wheat	2000	0.899	0.75	0.088	1	0.1	0.65	0.35
Rape seed	2000	0.100	0.77	6.914	1	0.48	0.8	0.26
Glass	2000	0.001	2.6	2595.554	1	0.52	1	0.1

δ_A is the density.

The dividers/methods in the worst group include, amongst others, all the shoveling methods and many of the methods which have really substantial losses, show large user-dependency, are (too) slow or have severe difficulties to end up with the correct target concentration or mass. It is, however, important to consider carefully the purpose of the method/device, so as to make the correct choice. In this context users should pay special attention to the parameters of importance in the specific situation.

4.3. Estimation and comparison of the Fundamental Sampling Error

The Fundamental Sampling Error (FSE) is the error that remains when the sampling procedure is rid of incorrect errors and faults. This means that FSE is the minimum sampling error that can be obtained in practice and it is inherent only to the material heterogeneity. For this very reason it is, of cause, method-independent. FSE can be calculated from a series of measurements as the difference between the estimate of the lot grade, a_S, and the actual lot grade, a_L (known in the present experiments):

$$\mathrm{FSE} = \frac{a_S - a_L}{a_L}$$

FSE for a given material can also be estimated beforehand using the so-called "Pierre Gy formula" [1,2]:

$$s^2(\mathrm{FSE}) = cfg\beta d^3\left(\frac{1}{M_S} - \frac{1}{M_L}\right)$$

- c is the constitution parameter expressed in g/cm^3 that accounts for the densities as well as the proportions of the constituents.
- f is a "particle shape factor" (dimensionless) describing the deviation from the ideal shape of a cube. A square will have $f=1$, a sphere $f=0.52$ and an almost flat disc $f=0.1$.
- g is a "size distribution factor" (dimensionless) describing the span of particle sizes in the lot. Default values are estimated by Gy and Pitard [1,2].
- β is a "liberation factor" (dimensionless) describing the degree of liberation of the critical component from the matrix. Totally liberated particles means $\beta=1$ and totally incorporated particles means $\beta=0$.
- d is the "top particle size", defined as the square-mesh screen that retains 5% of the material (dimension of length expressed in cm)—this does not necessarily correspond to the physical particle diameter, as in the case of "cylindrical" particles such as wheat.

The parameters listed in Table 2 were used to calculate FSE for the given materials used here.

The ratios shown in Figs. 32–34 should optimally be around 1.0, which would imply that the methods or devices only have a sampling error in the range of the Fundamental Sampling Error (FSE), implying very low deviation from minimum practical sampling error. Pierre Gy's estimate is

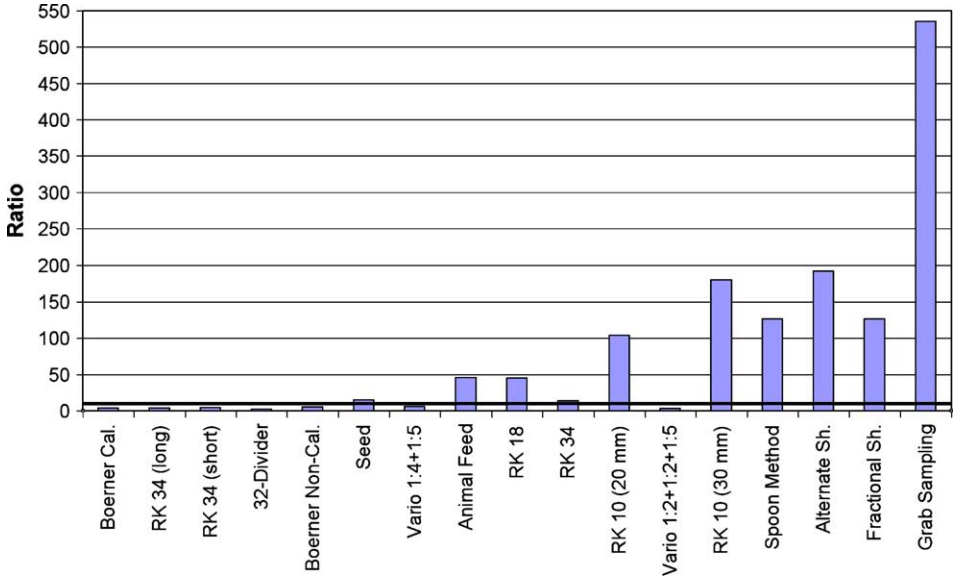

Fig. 32. Ratio between estimate of FSE and FSE from experimental procedure (for wheat). The horizontal line depicts the ratio of 10, indicating that all ratios lower than this is within an order of magnitude from the Pierre Gy formula estimate of FSE.

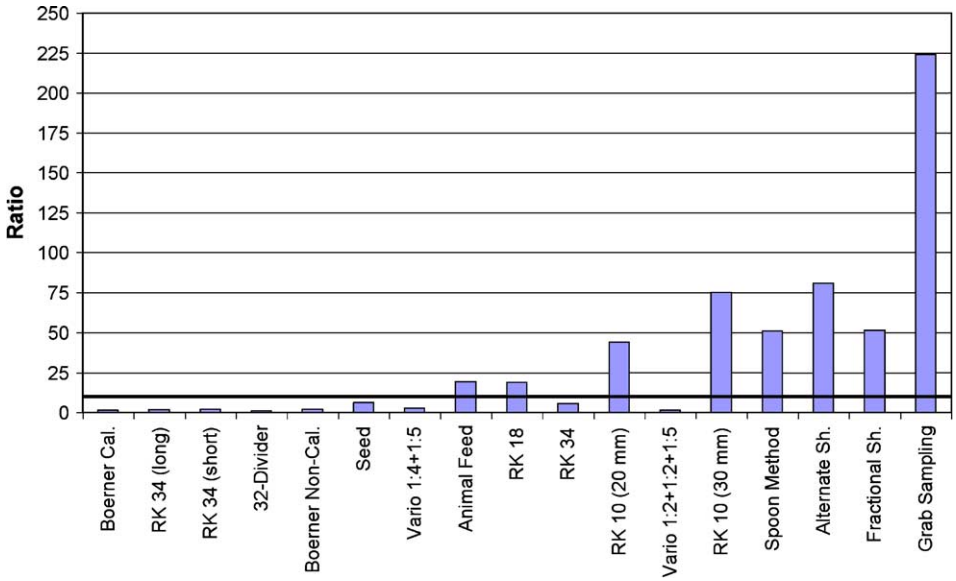

Fig. 33. Ratio between estimate of FSE and FSE from experimental procedure (for rape seed). The horizontal line depicts the ratio of 10, indicating that all ratios lower than this is within an order of magnitude from the Pierre Gy formula estimate of FSE.

meant to give the order of magnitude for the value of FSE; in this case the ratio should maximally be 10. This is marked as the flat line shown in Figs. 32–34. From the figures it can be observed that the estimates fit nicely with the experimental values for all acceptable methods, indicating that Pierre Gy's formula can be used for getting a rough estimate of FSE prior to any experimental procedure. At the same time, it further indicates the great overall performance of the best of the methods. It must, however, be stressed clearly that Pierre Gy's formula only yields an *estimate to an order of magnitude* of FSE, and must not be taken for an absolutely true value.

5. Conclusions

In order to achieve the best possible mass reduction it is crucial that the operator clearly analyses and defines the needs in a specific situation. In the overall characterization of the methods it has been decided to weigh all the characterizing parameters equally, even though this might be unreasonable for some applications in certain situations. We ask the readers to make their own modified conclusions from their specific needs; it will be very easy to consult Table 1 in this context. We have here emphasized the overall pooled characteristics of the methods investigated.

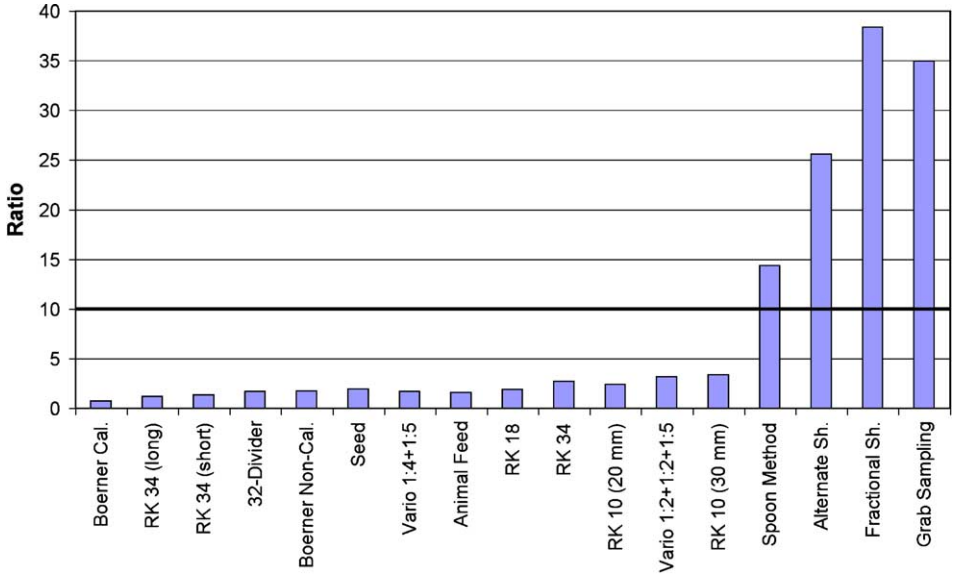

Fig. 34. Ratio between estimate of FSE and FSE from experimental procedure (for glass). The horizontal line depicts the ratio of 10, indicating that all ratios lower than this is within an order of magnitude from the Pierre Gy formula estimate of FSE.

Fig. 35. Grab sampling—the world's *worst* mass reduction/sampling method! If the lot material is heterogeneous and/or segregated (which is most often the case), grab sampling is the simplest and fastest way to get *heavily biased samples*. A miniature riffle splitter can easily be used instead—adding only seconds to the total preparation time, but several orders-of-magnitude to the representativeness. Grab sampling is to be totally avoided!

If all parameters can indeed be equally weighed, the following conclusions can be drawn.

The best overall methods for mass reduction are:

- Boener divider (cal.)
- RK 34 chutes (10 mm) short
- RK 34 chutes (10 mm) long
- Rotating 32-divider
- Vario Divider with splitting ratio 1:4 + 1:5

Of these methods riffle splitters are portable devices to be used both in the field or laboratory, while the latter two are heavy machines that can only be installed permanently for high speed reductions in permanent sampling stations or in laboratories. The Boerner Divider is heavy, though it also can be classified as portable if in a tight spot. All five methods perform excellently in both finding the correct target concentrations as well as having the nominal split-mass in the final samples. This compound criterion includes exactly what should rightly be characterized as the two most important parameters of a mass reduction device or method. Any differences concerning proper mass reduction between these five devices are minor and can be regarded as insignificant.

5.1. Total rejection of the world's most often used method—grab sampling

In general, all grab sampling and shoveling methods must be totally avoided; indeed grab sampling should never be used in practice—with the singular exception for thoroughly homogenized fine powders. It is a sad state of affairs that it is indeed a really fast, easy and cheap method since it "just happens" to be the absolute worst of all mass reduction methods. Fig. 35 tells its own story directly with the utmost clarity.

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