Representative Mass Reduction
A Critical Survey

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INTRODUCTION

The most common error of ill-reflected sampling is to focus on getting to the final sample volume much too early in the process. Instead of focusing on securing representative samples (which cannot be evidenced from the physical samples themselves) TOS, Theory of Sampling, stipulates that only a properly designed and controlled sampling process can facilitate this. 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. This places stringent demands on the sampler (or the sampling instrument), for instance if the sample is used for chemical analysis, where typically only a gram, or a fraction hereof, is required. There is often a (very) long way from the size of the lot, *via* the primary sample to the final analytical sample volume or mass. Typically mass reductions of the order of 1:1000 to 100.000 (or more) are encountered. It is therefore of outmost importance that all sampling processes involve *representative* mass reduction. Unfortunately many hardware designers and manufacturers look at mass reduction as a pure volume/mass reduction in terms of weight *per se*. It's quite another thing to be concerned about the degree of representativity of the reduced mass fractions – which is our purpose here.
The issues surrounding how to do the *primary sampling*, as this is amply covered in the basic sampling literature [Gy, Pitard, Smith].

This contribution focuses on a comparison of sixteen methods and devices extensively used today for mass reduction in industry and in the laboratory, which have been tested and assessed with regard to a number of characterizing parameters, among which the most prominent are *accuracy* and *reproducibility* (precision). These two constitute the definition of *representativeness* [Gy, Pitard].

We are interested in the quality of both the average composition estimates resulting from mass reduction operations as well as 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 final sample mass, loss of material, time consumption, user dependency and device cleaning requirements are included in the overall survey presented here.

Mass reduction is NOT just a mere materials handling technique – it is a critical and sensitive preparation step that CANNOT be disregarded in sampling and analysis!

MATERIAL SYSTEM

Which kind of material system could be optimal for comparison purposes? Should the composition of the system reflect only one specific situation with a relatively smaller range of potential applications fields, or should one strive for as general a material system as possible?

These requirements for the system were delineated:

- It must reflect very different concentration levels
- It must be susceptible to segregation
- It must show a tendency towards "rebounding" behavior, to simulate natural, "difficult to handle" systems (rape seed and glass below)

For the extensive comparison series the following system was chosen (percentages given in weight-by-weight):

89.9 %	**Wheat grain**	(approx. 2 by 6 mm *cylinders*)
10.0 %	**Rape seed**	(approx. 2 mm diameter *spheres*)
1.1 %	**Glass beads**	(approx. 1 mm diameter *spheres*)

This system setup was also influenced by the fact that these components were easy to "analyse" (separation by a sieving system).

EXPERIMENTAL PROCEDURE

- The constituents were carefully weighed and mixed to have exactly the same composition for every experimental run. The total mass of material was always 2 kg.
- A mass reduction (or series of reductions) was performed, resulting in a final sample mass of 100 or 125 g, depending on the nature of the method or device used.
- The final sample *and* the remnant material were separated into their constituents by careful sieving (Figure 1).

Figure 1. The home-built sieving system.

- All constituent fractions (in both sample and remnant portions) were weighed, from which compositions, total masses, loss etc. were calculated.
- This procedure was repeated 20 times, in two blocks of 10 (two different operators) for all methods/devices.

RIFFLE SPLITTING

A riffle splitter consists of a (preferably) large number of parallel chutes leading, in alternating fashion, to two opposite receptacles. The chutes must be of equal width, shape and make, and equal in number. The chutes have to have a certain minimum width which depends on the particle size, in order to prevent blocking (large particles) or bridging (powders). A rule of thumb stipulates a minimum width of 5 mm or 3 times the largest particle diameter.

The material to be split is fed over *all* the chutes *evenly* by a feeding tray that has exactly the same width as the splitter length.
Selection of receptacle containing the final sample, or the material to be further split, *must* be made randomly.

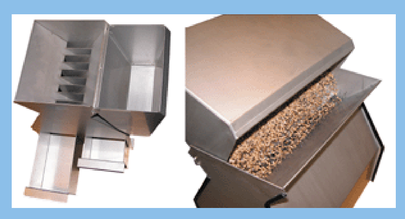

Figure 2. Example of a riffle splitter (Nr. 4) – too few chutes!

In the present work six different riffle splitter types were tested. Figures 2 and 3 show some of the splitters involved. During the experimental runs several optimizations of existing devices and the design of a new device took place. The splitters tested were named according to design for easier distinction as follows:

2. Animal feed splitter
3. Seed splitter
4. RK 10 chutes / 20 mm chute width
5. RK 10 chutes / 30 mm chute width
6. RK 18 chutes / 16 mm chute width
7. RK 34 chutes / 10 mm chute width

Figure 3. Further examples of riffle splitters.

Left (no. 1), middle (no. 2) and right (no. 6)

REVOLVING DIVIDERS

Revolving splitters are based on the same principle as riffle splitters and the Boerner divider. The revolving feeding funnel 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, depending on the settings and design of the device. The precision of the device depends on the rotating speed as well as the influx velocity of the material through the feeding funnel and the grain-size contrast of the material. The splitting principle is that every second chute contributes to one of two alternative collecting reservoirs – or to more than two, resulting in *variable split ratios*.

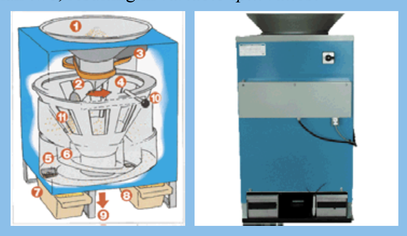

Figure 5. The Vario Divider. Revolving splitter with variable chute width.

The investigated revolving dividers were the Vario Divider with variable chute-width (computer controlled), Figure 5, and the so-called Fixed 32-divider, Figure 6. In the figure above the working principle is outlined in detail. The revolving feeding funnel (2) distributes the material over the chutes (11), which have variable width. The model shown here actually produces two samples (7 and 8) while discarding the remnant material (9). It is just a matter of connecting the different streams in order to design a desired split ratio. The Fixed 32-divider is slightly different. In the experimental series a plastic bag was mounted on all 32 outlet tubes, and two of these, the first chosen randomly - the second opposite hereof, were combined into a final 1/16 split of 125 g.

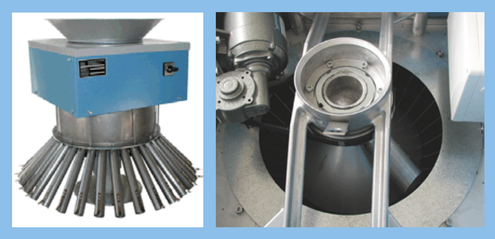

Figure 6. The Fixed 32-divider. 32 fixed, equal-sized radial chutes leading to separate outlet tubes. Optimal for preparing e.g. ring-test samples or for splitting large material volumes. Right: revolving inlet tube and radial chutes.

BOERNER DIVIDER

The Boerner divider is often regarded as a standard in mass reduction and has been extensively used, e.g. within seed testing.
The device receives the mass to be sampled through the hopper. Since material is flowing downwards directly onto the top of the dividing cone, statistically it should spread out evenly in all azimuth directions. At the bottom of the cone the material is led through a number of alternative chutes (44 in the investigated device), which are so connected that every other leads to two different receptacles, yielding two half-splits. Figure 4 show the investigated divider, both *in toto* and in a zoom of the cone "in action". Two different runs were performed with the Boerner divider, a calibrated and a non-calibrated model.

Figure 4. The Boerner divider with 44 chutes

(right – *zoom in* of the dividing cone "in action").

SHOVELING METHODS

A traditional category – shoveling methods - consists of four different, more or less frequently used methods. Shoveling methods are generally fast and easy to use, but the results are very nearly always inferior (to distinctly bad), unless the efforts laid down are unreasonable large for many practical purposes.

The Spoon Method is much used within seed testing organizations. The method consists of spreading the material to be sampled carefully in a tray in an "s-like" pattern, layer by layer. At least five small scoops of material are extracted (depending on the size of the final sample) by placing a knife and scooping up the material all the way to the bottom of the tray, Figure 7.

Figure 7. The Spoon Method. The lot is poured in an "s-like" pattern and scooped up in sub-samples which are combined into a final composite sample.

Alternate shoveling consists of scooping the material and depositing it in an alternating fashion between two reservoirs. The larger the number of scoops, the better the reduction. Involuntary tendencies like scooping from the same spot have to be avoided.

Figure 8. Alternate shoveling.

Figure 9. Fractional shoveling

Fractional shoveling is based on the same principle as alternating shoveling. The number of reservoirs can be anything more than two however. In our experimental series four and five reservoirs were used, in two successive reductions, yielding a final sample of 100 g.

Grab sampling is the "enfant terrible" of sampling. It amounts to taking a single scoop from any easily accessible part of the lot. This violates the fundamental sampling principle: all potential increments must have an equal, non-zero, sampling probability. Grab sampling yields the largest analytical variation (if replicated): To be avoided at all costs!

Figure 10. Grab sampling.

CONCLUSIONS

From the experiments the following conclusions can be drawn:

- Extreme care must be taken when choosing the method or device for mass reduction since *great* differences exist between their rated performances. RK 34 & fixed 32-divider are superior.
- Pilot-experiments should always be performed to determine whether a method or device performs satisfactorily for a certain purpose, especially for new materials.
- Well-designed and engineered devices like *riffle splitters* (preferably with large number of chutes), *Boerner dividers* or *revolving splitters* nearly always provide reliable and representative mass reductions. However attention must be paid to design details, since even the smallest errors in design or deviation from the principle of correctness [Gy, Pitard, Smith] can result in a severe sampling bias for the final samples.
- Methods like grab sampling and other shoveling methods can rarely, if ever provide reliable results. They must be performed with very great care and insight and should never be used unless no other alternative exists.

RESULTS

Here we present only a *representative sample* of our results.

The most important parameter characterizing a mass reduction device or method is the **representativeness**, r, since it includes both accuracy and precision:

$$r^2(SE) = m^2(SE) + \sigma^2(SE) \qquad SE=(a_i-a_L)/a_L$$

a_i = Grade of Component i in the Sample/Grade of the Lot

Figure 11 show the accumulated sum of representativeness for all three constituents (wheat, rape seed and glass). Lower values indicate better methods or devices. It is clear that glass is the main contributor to the sum since the concentrations varies heavily in the final samples (trace concentration 0.1 % w/w only). The same tendency is clear for all three constituents: Grab and shoveling methods provides absolutely the worst reductions! Riffle splitters (high number of chutes), revolving splitters and the Boerner divider clearly provide the best mass reduction methods/devices; Boerner divider, RK 34 and the Fixed 32-divider appear superior.

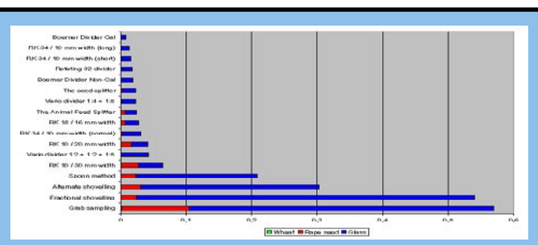

Figure 11. Sum of representativeness for wheat, rape seed and glass.

Figure 12 shows the percent deviation from the designated mass of the final sample (100 or 125 g). Grab and shoveling methods again perform less satisfactory than other methods.

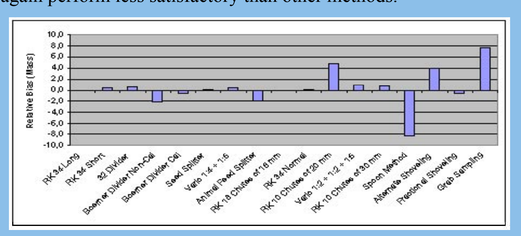

Figure 12. Relative bias of sample mass (%-wise difference from 100 or 125 g)

All the investigated methods and devices are rated on a one-to-one weighed basis with regard to parameters: compliance with true lot composition and with designated mass, degree of loss of material, ease of device cleaning and maintenance, performance initialization time, operation time and user-dependability. These diverse ratings are summarized in Table 1.

Table 1. Mass reduction method ratings. Green indicates "excellent", - grey indicates "acceptable" and red indicates "unacceptable" overall performance.

	RK 34 / 10 mm (long)	RK 34 / 10 mm (short)	32 divider	Boerner div. (non-cal.)	Boerner div. (Cal.)	The seed splitter	Vario 1:4 + 1:5	Animal feed splitter	RK 18 / 16 mm width	RK 34 Normal	RK 10 / 20 mm width	Vario 1:2 + 1:2 + 1:5	RK 10 / 30 mm width	Spoon method	Alternate shoveling	Fractional shoveling	Grab sampling
Composition	+	+	+	0	+	+	+	0	+	+	0	0	0	-	-	-	-
Mass Loss	+	+	+	-	-	+	+	+	+	+	-	+	+	-	-	-	-
Cleaning	0	0	0	0	0	+	+	+	0	0	0	+	0	-	+	-	+
Initialization	+	+	+	0	0	+	+	0	+	+	0	+	0	0	+	0	+
User dep.	0	0	+	0	0	0	0	0	0	0	0	+	0	0	-	-	+
Time	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Score	**5**	**5**	**5**	**2**	**3**	**4**	**5**	**2**	**4**	**4**	**1**	**4**	**3**	**-3**	**-2**	**-3**	**1**

The methods divide into three groups: Excellent (green), mainly acceptable (grey) and unacceptable (red). One-to-one weighing of the parameters may not appear correct, but weights can easily be altered so that specific parameters can be granted different individual influence. RK 34 and the fixed 32-divider reign supreme.

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