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TOS forum

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It's time again…

After the successful 8th World Conference on Sampling and Blending, WCSB8, May 2017, Perth, Australia, a year has rushed by. With a nominal publication frequency of two issues of *TOS Forum* per year, it is indeed time for the next issue.

This issue is a blessed mixture of old and new, small and big items of interest.

The opening feature addresses a very difficult issue: how to sample Municipal Solid Waste (MSW)? MSW is an example of just about the most heterogeneous material imaginable. By its nature *household waste* is extremely variable in overall composition (day-to-day, city-to-city, country-to-country) and will also change its composition temporally, as a function of a yearly cycle. But knowing this is a vital prerequisite for effective implementation of MSW management facilities, which is an issue of societal importance that will only grow. From a Theory of Sampling perspective, this issue adds to the full scope of representative sampling, because it is of paramount importance that the lot material is put through a thorough sorting before TOS has even a first chance to be bias-free. **Philippe Wavrer** (CASPEO) has penned a fascinating account of a large-scale French endeavour.

Next, the chairman of WCSB8 (2017), **Ralph Holmes**, presents his comprehensive report from our previous world conference. Besides the scientific meeting and its achievements, duly published in the proceedings which is in the very experienced hands of the AusIMM, WSCB8 was a *special event* in that a highpoint was the launching of the **International Pierre Gy Sampling Association (IPGSA)**. For the first time since the first WCSB1 (2003), our community now has the form of a scientific organisation which will handle organisational matters for which such a formal entity is necessary. The historic background for the proposal to inaugurate a formal scientific organisation was outlined in the previous *TOS Forum* issue—and the first president of the IPGSA takes this development further in the present report. The inaugural IPGSA Council, and its advisory committee, is duly announced here as well. In a coming issue we will present the Council further, complete with biographies and declarations-of-intent, all so that we can all fully appreciate the collective we have now elected to run our administrative, organisational and other matters.

This issue focuses on the 2017 recipient of the Pierre Gy Sampling Gold Medal, **Richard Minnitt**. *TOS Forum* brings you his inaugural "**Pierre Gy Oration**", which could not have been in better hands. In addition, you will also find another of Dick's contributions to WCSB8, this one on "The costs of Sampling Errors and Bias in the Mining Industry". We bring both these features in an exciting new format, which we hope will find acceptance.

There follows a feature which at first *may* appear slightly off-Broadway, entitled: "**The 2010 Eyjafjellajökul volcanic ash sampling experiment: factors and conditions affecting field sampling variability**", but which in essence focusses on a distinctly under-appreciated aspect of primary sampling, particularly within the natural sciences, characterised by the fact that there is absolutely no control over the primary lot and its origin. This is demonstrably different from the common situation within technology and industry, which calls for *some* care before hitting the field. This feature actually depicts the scientific highpoint of a 60-year birthday party!

Finally, last but by no means least, the updated brochure for the next World Conference on Sampling and Blending (WCSB9), 6–9 May 2019, Beijing, China, is presented. The final deadline for abstract submission is 9 November. This is a welcome opportunity to reach out to all of us one last time: **DO NOT MISS SUBMITTING AN ABSTRACT, OR REGISTRERING FOR WCSB9.**

See you all in Beijing in 2019!

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A unique field sampling experiment on the volcanic ash from the 2010 Eyjafjellajökull eruption. See page 22 for the full story.

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Theory of Sampling (TOS) applied to characterisation of Municipal Solid Waste (MSW)—a case study from France[a]

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Knowing the composition of household waste is a prerequisite for effective implementation of municipal solid waste (MSW) management facilities. To meet increasing regulations, facilities in terms of collection, sorting and treatment are becoming more sophisticated and expensive: performance reliability partly depends on a valid, representative knowledge of waste composition. In France, the current method of characterisation of household waste is MODECOM™, a guide to organise and manage analysis campaigns with the primary objective of evaluating the recyclable or the packaging material content of waste, or to determine the variations and characteristics related to the nature of housing, for example. Implementation of this methodology leads to primary MSW samples, which are successively screened and sorted into a set of standard categories. Although it is possible to determine the composition of household waste in this fashion, at the end of these operations looms the question of its accuracy. Even if the mass of fully sorted MSW samples (usually around 500 kg) may seem high, this is actually extremely small compared to the total lot from which it was sampled (several hundreds of tons, sometimes much more). The Theory of Sampling of particulate materials (TOS), as initially developed by Pierre Gy in the context of the mineral industry, is quite applicable also to household waste. In particular, it allows an estimate of the Fundamental Sampling Error (FSE) to be calculated for each of the sorted categories. From real-world examples of French MSW characterisations, this contribution shows which data are needed and how the FSE formulas are implemented, illustrating how it is possible to ascribe individual total error estimates for each category. This general overview will help local implementation efforts.

Introduction

Knowing the composition of Municipal Solid Waste (MSW) is a key element of waste management policy for local authorities. This knowledge is essential to anticipate change and to set up the treatment necessary (procedures, equipment) for optimised extraction of the valuable parts of the waste.

However, the composition of household waste may vary in **space**, e.g. from one administrative district to another, and may depend on the geographical region, the type of habitat etc. And it may also vary in **time** according to the season or the evolution of consumption practices.

Characterisation of waste necessarily requires a sampling phase prior to analysis. After this step, decisions that often will have significant consequences will be taken in terms of risk assessment, protective measures, fees, selection and magnitude of treatment processes. Depending on the specific nature of the waste and on the diligence of its characterisation, the risk of providing an incorrect advice may be greater or smaller, especially as analysis is usually only performed on sub-samples of severely reduced size. The evergreen question for practically all commodities and materials, waste no exception, is then: are the samples analysed *representative* of the whole waste lot targeted? And how can this important question be answered? The Theory of Sampling to the fore…

Characterisation of municipal solid waste

European countries have developed several municipal solid waste (MSW) characterisation methods.[1] For example:

- ARGUS (in Germany),
- IBGE (in Belgium),
- EPA (in Ireland),
- MODECOM[b] (in France).

Although each of these addresses its own specific national requirements, they all conform to a common approach: after a first sampling step, the different types of waste contained in the sample are sorted into fractions and categories.

The French approach

In the early 1990s, there was a notable lack of knowledge about the composition of household waste at the national level and a lack of a reference method for comparing data between municipalities or regions managed by different local authorities. In order to address these shortcomings, a programme for characterisation of household waste at the national level was carried out in 1993 by ADEME, the French Environment and Energy Management Agency. Since no method nor reference data about waste existed at that time, it was necessary first to develop a methodology based on feedback from French and foreign sources. This became the MODECOM, acronym for “MéthOde DE Caractérisation des Ordures Ménagères” or Method for Characterisation of Domestic Waste.[2] It has been transcribed as standards by AFNOR, the French Association for Standardization.[3,4]

Representing the real starting point for estimating the composition of household waste at the national level, the MODECOM methodology made it possible to better understand household residual waste streams on French territory.

This methodological tool is still used today, although in a substantially modified version. It was implemented in 1993 during the first national campaign for characterisation of domestic waste, in order to achieve an inventory of the “average composition of the waste bin of a French inhabitant”. Fifteen years later, in 2007–2008, a second national characterisation campaign was carried out, still based on

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[a] This paper unfortunately did not make it to be presented at WCSB8 due to cancellations. *TOS Forum* is delighted to be of service.

[b] MODECOM is a registered trademark of ADEME.

MODECOM, to estimate “variations of the composition of typical household wastes” and to adapt the waste management systems accordingly. A third national campaign is currently underway; the results are expected in 2019.

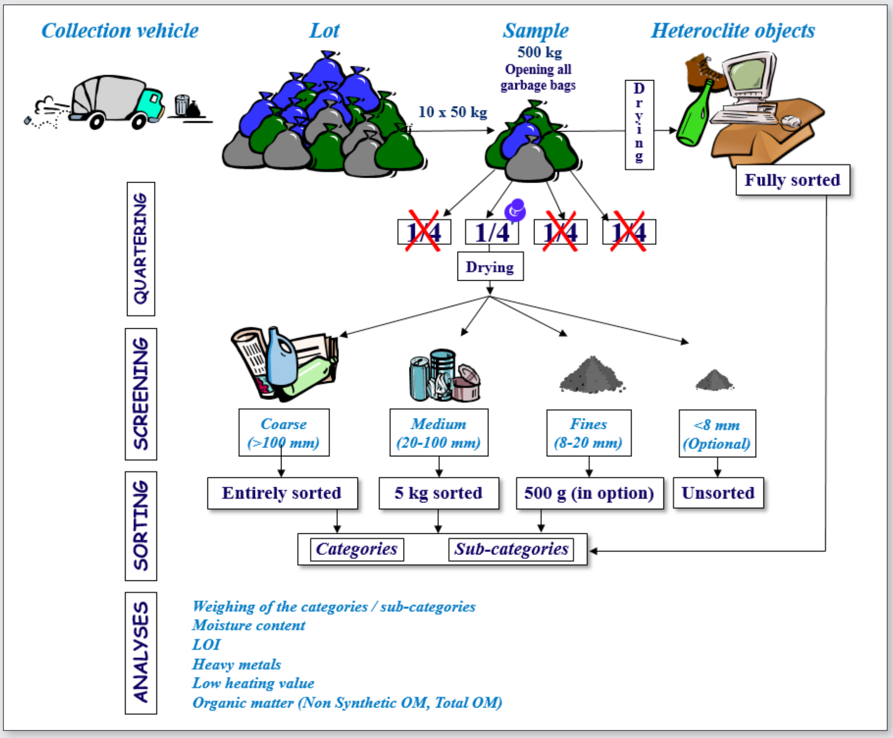


Figure 1. MODECOM sampling and sorting operations.

Figure 2. The lot of MSW before any sampling and characterisation operations.

Implementation and results of the MODECOM methodology

As it was designed, the MODECOM methodology consists of five major operations, or phases.

1) Preliminary inquiry, designed to collect all data required to *organise* the analytical survey. It may be of interest to subdivide, to stratify, a given area into different sectors, for example according to geographic zones, population districts, type of habitat, economic activity zones amongst others.
2) As MODECOM is based on characterisation of MSW from collection vehicles, the second step consists in selection of which collection vehicles to be sampled (primary sampling). For each stratum defined in the first step, collection vehicles are randomly selected (stratified random selection of collection vehicles assuming equi-probability within strata) based on random numbers, e.g. relating to the order of arrival of the vehicles to the treatment plant. Each vehicle should not contain less than 2 tons of waste. As vehicles are randomly selected within each stratum, every ton of collected waste has the same probability of being drawn; this assured compliance with TOS’ Fundamental Sampling Principle.
3) Formation of approximately 500 kg *composite samples* by random selection of 10 increments (of approximately 50 kg each) from the contents of each selected collection vehicle. Increment selection is also here based on random numbers, this time relating to the spatial ordering of potential increments of 50 kg collectively making up entire load contents of the selected vehicle.
4) Concerning sorting, several possibilities are offered according to the specific objectives of the intended characterisation. The one that is taken into account here is the standardised dry sorting method[5] (Figure 1).
    4.1) Opening of all household, and other types of *garbage bags*, in the 500 kg samples (Figure 2), following which all *heteroclite objects* are removed; these will be sorted separately from the remainder of the sample. A heteroclite object is defined as a “single fragment that contributes significantly to heterogeneity by its mass, its volume or its exceptional nature”.
    4.2) Quartering of the rest of the sample (Figure 3).

4.3) Drying of both the extracted heteroclite fraction and the remaining sub-sample after quartering at 70 °C for 5 days.

4.4) Screening using sieves (or *trommel*) with 100 mm, 20 mm and 8 mm apertures.

4.5) Sorting of coarse elements (>100 mm) and partial sorting of medium-size elements (20–100 mm). Optionally, 8–20 mm fine elements may be also partially sorted.

The contents of the screened and dried samples are hereafter sorted into at least the 12 basic categories of MODECOM (Table 1 and Figure 4). Depending on the objectives of the characterisation survey, some categories may be further sorted into sub-categories amongst others.

5) Laboratory analyses in order to determine standard parameters, e.g. moisture content, lost on ignition (LOI), heavy metals content,[6] low heating value, organic matter content (in particular non-synthetic organic matter content).

The objective of MODECOM was originally to determine the characteristics of MSW produced at the level of an administrative area managed by *local authorities*. Nevertheless, it is also used to determine the composition of MSW at the lower level corresponding to a single waste treatment plant to establish material balances, for example. In this case, only steps 3–5 are involved, i.e. formation of composite sample, sorting and the laboratory analyses. Each of these steps is carried out following the dedicated standard.[7,4,5]

Characterisation results can be presented in several ways, depending on which categories, sub-categories and particle sizes are considered. Classically, the composition of MSW is presented using only the 12 basic categories (Figure 5).

From a rigorous point of view, this compositional assessment is strictly only valid for the single 500-kg composite sample which has been sorted. However, the results from this will be *extrapolated* to the whole waste lot from which this primary sample was taken. This is a critical issue regarding MODECOM—is this canonical sample size adequate for all purposes?

Table 1. Nomenclature of standard categories and sub-categories in MODECOM.[c]

Categories	Sub-categories
Putrescible waste	Food waste
	Unconsumed food products
	Garden waste
	Other putrescible waste
Papers	Packaging
	Newspapers, magazines, brochures
	Printed advertising matter
	Office papers
	Other papers
Cardboards	Flat packaging cardboard
	Corrugated packaging cardboard
	Other cardboard
Composites	Cardboard composites packaging
	Small appliances
	Other composites packaging
Textiles	Textiles
Health care textiles	Health care textiles, hygienic fraction
	Health care textiles, soiled papers fraction
Plastics	Polyolefine films (PE / PP)
	PET jars and bottles
	Polyolefin jars and bottles
	Other plastics packaging
	Other plastics
Unclassified combustibles	Wood packaging
	Other combustibles
Glass	Colourless glass packaging
	Colour glass packaging
	Other glass
Metals	Ferrous metal packaging
	Aluminium packaging
	Other ferrous metal waste
	Other metal waste
Unclassified incombustibles	Unclassified incombustibles packaging
	Other unclassified incombustibles
Dangerous waste	Chemical products
	Fluorescent tubes and energy saving lamps
	Batteries and accumulators
	Other dangerous waste
Fine elements (–20 mm)	Fine elements with a size ranging from 8 mm to 20 mm
	Fine elements smaller than 8 mm (round mesh)

[c] This nomenclature is the one used for the 2007 national campaign. The list and definition of the sub-categories have later been substantially modified for the current national characterisation campaign to take into account the evolution of the MSW and the changing objectives of the campaign.

Application of the Theory of Sampling (TOS) to MSW

MSW is a solid material with a very obvious *heterogeneous* composition. However extreme this maybe, it is fully possible to apply Pierre Gy's Theory of Sampling, TOS,[8] without any problem. For the moment disregarding the effects that reflect geographical or seasonal variations (which are fairly easy to compensate for by focused application of MODECOM), the following calculations focus on the *constitution heterogeneity* of MSW (Compositional Heterogeneity, CH) which is always high. The constitution heterogeneity (CH) is a result of the varying proportions and physico–chemical properties of the constituent *elements* (units) of the MSW, which generates the Fundamental Sampling Error (FSE). TOS allows to estimate the Fundamental Sampling Error (FSE) variance starting from the heterogeneity model (the compositional MSW characterisation expressed as the standard 12 categories), with respect to the different analytical parameter to be measured.

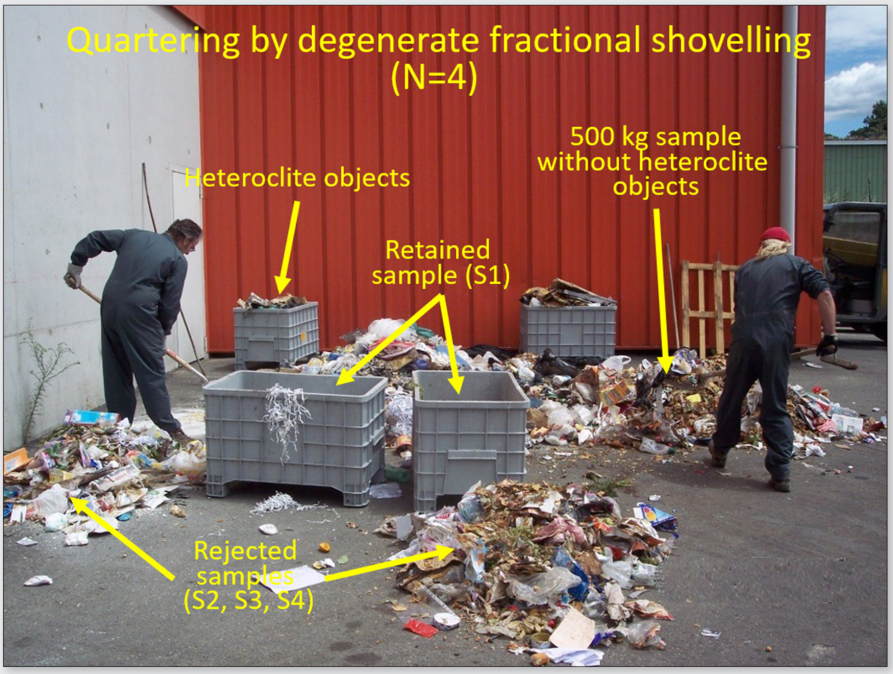


Figure 3. Quartering of the primary sample after removing of the heteroclite objects (step 4.2).

Fundamental Sampling Error (FSE) of the proportions of MSW categories

When sampling MSW, the randomly selected units are *particles* of a very disparate nature. But these particles can be *classified* into families of *similar* particles, mainly regarding their size and composition. This is what is facilitated at the different MODECOM sorting stages: particles are sorted into sizes, categories and sub-categories. For example, particles of paper are considered to be paper with a content of 100 % (and 0 % of **any** other constituent)—and similarly for all other categories/sub-categories and constituents.

It is now *assumed* that, after sorting, the sizes, masses and compositions of the

Figure 4. Example of coarse elements (>100 mm) after sorting.

sampled particles display sufficiently narrow ranges so as meaningfully to constitute quasi-homogeneous *families* (a standard assumption in TOS). The measured parameter is the *family proportion* itself. This parameter is not distributed in all the material, but confined to one and the same family. In this case, the particles are called *simple particles.*

The relative variance $\sigma^2(FSE)$ of the Fundamental Sampling Error for the constituent composition of the lot is given by Pierre Gy's formula for simple particles (consisting either of 100 % or 0 % of the constituent in question) (Equation 1).

$$\sigma^2(\mathrm{FSE}) = \left(\frac{1}{M_s} - \frac{1}{M}\right)\left[\left(m_c \frac{1-2t_c}{t_c}\right) + \sum_{i=1}^{n} t_i m_i\right] \tag{1}$$

With:

- σ^2(FSE) the relative variance of the Fundamental Sampling Error for the proportion of the family *c*
- M_s the sample mass
- M the mass of the initial batch (lot) to be sampled
- t_c *the mass proportion of family* c *in the sample*. This is the parameter that we attempt to determine through appropriate sampling
- t_i the mass proportion of family *i* in the sample
- m_c the mean unit mass of one particle of family *c*
- m_i the mean unit mass of one particle of family *i*

Experience shows that this sample mass, 500 kg, recommended by the MODECOM procedure, has been observed using this formula as being able to reach a *sufficient level* of representativeness for *most families* with a *reasonable* and *manageable* sample size, see references.

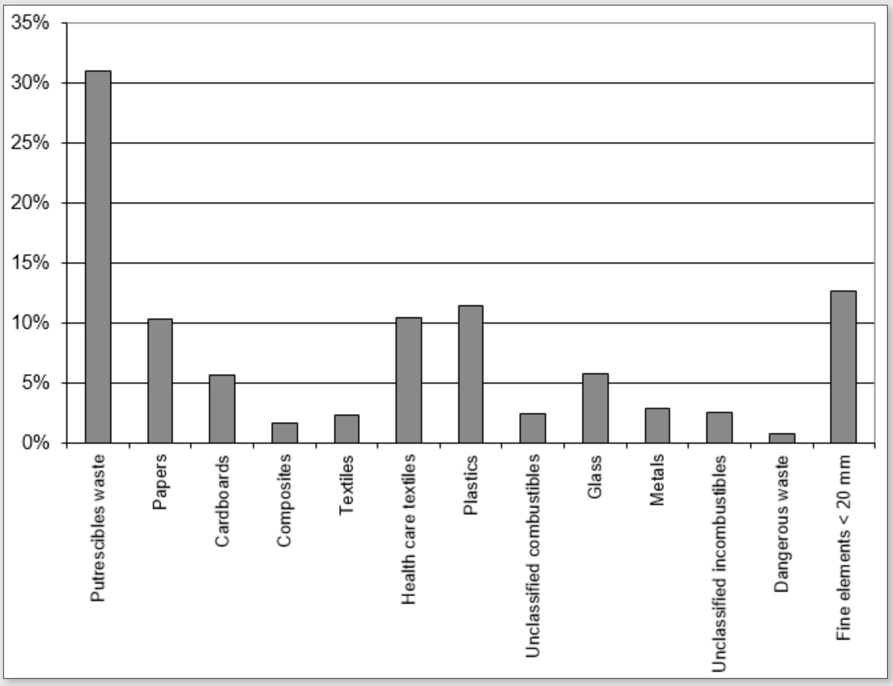


Figure 5. Example of a global MSW composition accounting after MODECOM sorting.

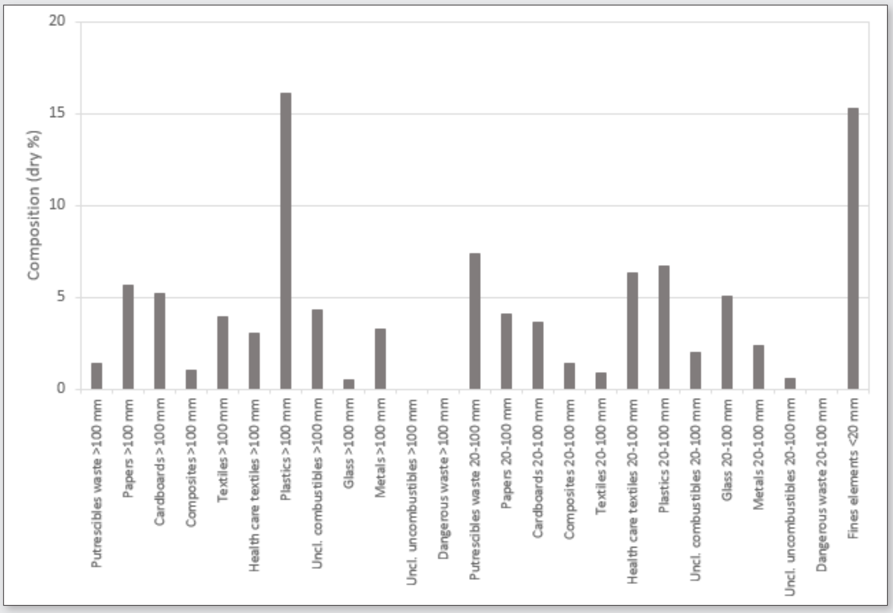


Figure 6. Composition of MSW from the French case study.

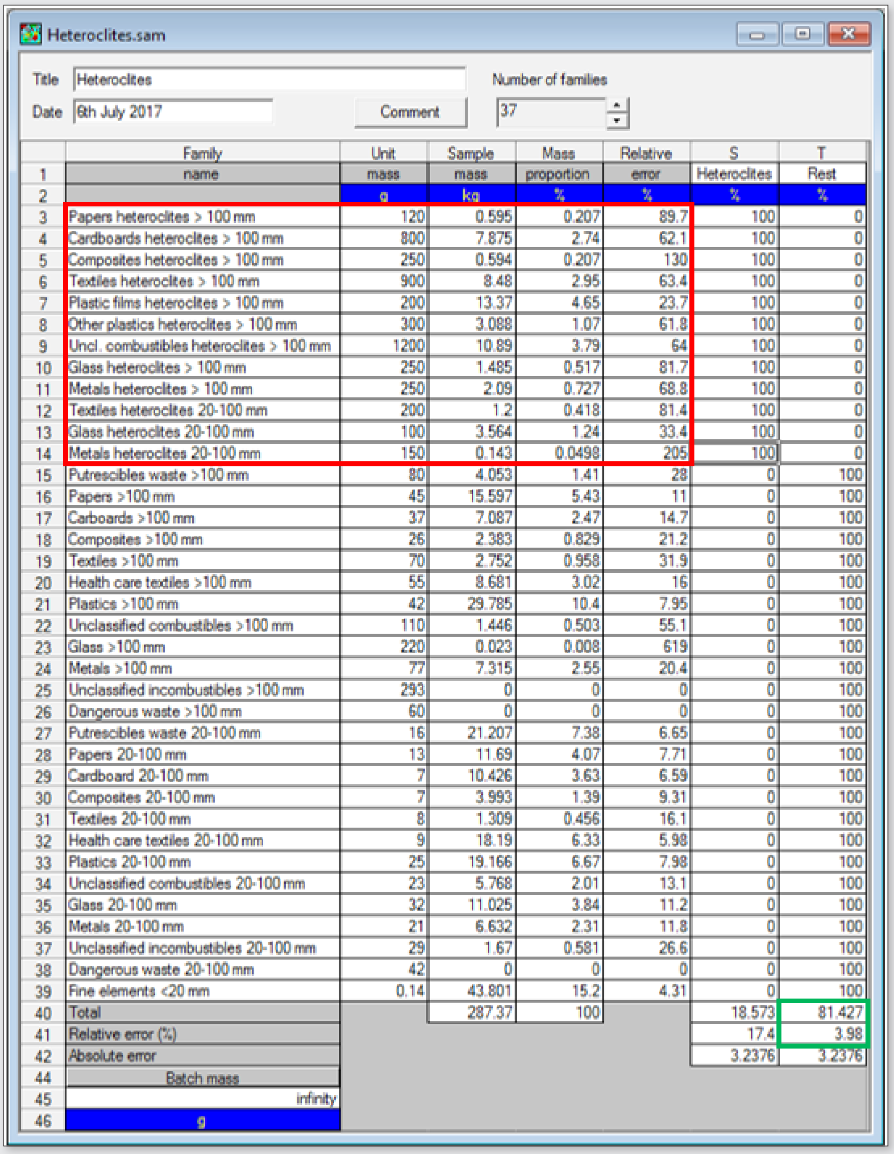
Determination of mean (average) unit masses

The mean (average) unit mass is a key factor which can be difficult to determine.[9,10] In the case of MSW, estimation of the unit masses by calculation, using size, density and shape factor of particles, is inappropriate and can be very inaccurate because of the extreme heterogeneity in MSW. The mean unit mass of each category/sub-category can alternatively be obtained by weighing the entire sorted family and dividing the resulting weight by the number of constituent particles. It is important to weigh a sufficiently large number of particles randomly, selected from *each* sorted family: 200 items is considered to a minimum.

Even if this operation **can** be performed for each waste characterisation, it is very time consuming. Some surveys for the determination of the average unit masses per category/sub-category were carried out at the national level.[11] More local and time-limited determinations have also been carried out in the frame of medium or large scale MSW characterisations. Databases gathering the average unit weights of the different household waste categories / sub-categories could therefore be aggregated and could be used for the determination of the fundamental sampling error following the above approach.

Example: a case study from France

To illustrate the approach, we consider here MSW treated in a biological treatment plant



Title: Heteroclites
Date: 6th July 2017
Number of families: 37

	Family name	Unit mass	Sample mass	Mass proportion	Relative error	S Heteroclites	T Rest
2		g	kg	%	%	%	%
3	Papers heteroclites > 100 mm	120	0.595	0.207	89.7	100	0
4	Cardboards heteroclites > 100 mm	800	7.875	2.74	62.1	100	0
5	Composites heteroclites > 100 mm	250	0.594	0.207	130	100	0
6	Textiles heteroclites > 100 mm	900	8.48	2.95	63.4	100	0
7	Plastic films heteroclites > 100 mm	200	13.37	4.65	23.7	100	0
8	Other plastics heteroclites > 100 mm	300	3.088	1.07	61.8	100	0
9	Uncl. combustibles heteroclites > 100 mm	1200	10.89	3.79	64	100	0
10	Glass heteroclites > 100 mm	250	1.485	0.517	81.7	100	0
11	Metals heteroclites > 100 mm	250	2.09	0.727	68.8	100	0
12	Textiles heteroclites 20-100 mm	200	1.2	0.418	81.4	100	0
13	Glass heteroclites 20-100 mm	100	3.564	1.24	33.4	100	0
14	Metals heteroclites 20-100 mm	150	0.143	0.0498	205	100	0
15	Putrescibles waste >100 mm	80	4.053	1.41	28	0	100
16	Papers >100 mm	45	15.597	5.43	11	0	100
17	Carboards >100 mm	37	7.087	2.47	14.7	0	100
18	Composites >100 mm	26	2.383	0.829	21.2	0	100
19	Textiles >100 mm	70	2.752	0.958	31.9	0	100
20	Health care textiles >100 mm	55	8.681	3.02	16	0	100
21	Plastics >100 mm	42	29.785	10.4	7.95	0	100
22	Unclassified combustibles >100 mm	110	1.446	0.503	55.1	0	100
23	Glass >100 mm	220	0.023	0.008	619	0	100
24	Metals >100 mm	77	7.315	2.55	20.4	0	100
25	Unclassified incombustibles >100 mm	293	0	0	0	0	100
26	Dangerous waste >100 mm	60	0	0	0	0	100
27	Putrescibles waste 20-100 mm	16	21.207	7.38	6.65	0	100
28	Papers 20-100 mm	13	11.69	4.07	7.71	0	100
29	Cardboard 20-100 mm	7	10.426	3.63	6.59	0	100
30	Composites 20-100 mm	7	3.993	1.39	9.31	0	100
31	Textiles 20-100 mm	8	1.309	0.456	16.1	0	100
32	Health care textiles 20-100 mm	9	18.19	6.33	5.98	0	100
33	Plastics 20-100 mm	25	19.166	6.67	7.98	0	100
34	Unclassified combustibles 20-100 mm	23	5.768	2.01	13.1	0	100
35	Glass 20-100 mm	32	11.025	3.84	11.2	0	100
36	Metals 20-100 mm	21	6.632	2.31	11.8	0	100
37	Unclassified incombustibles 20-100 mm	29	1.67	0.581	26.6	0	100
38	Dangerous waste 20-100 mm	42	0	0	0	0	100
39	Fine elements <20 mm	0.14	43.801	15.2	4.31	0	100
40	Total		287.37	100		18.573	81.427
41	Relative error (%)					17.4	3.98
42	Absolute error					3.2376	3.2376
44	Batch mass						
45	infinity						
46	g						

Figure 7. Relative Fundamental Sampling Errors at 95 % confidence per category of heteroclite objects for the 287 kg of sorted dry MSW sample.

in a city in North-West France. The selection of collection vehicles and composite sampling of the MSW from each has been implemented according to the MODECOM methodology described above. As a result, a sample of 512 kg was aggregated. The sorting was performed following the dry method (Figure 1) considering the 12 basic categories (Table 1) for both coarse fraction (>100 mm) and medium-sized fraction (20–100 mm). Fine elements <20 mm have not been sorted but are still considered as a category. After drying, the mass of the sample was 287 kg. The dry composition of the MSW, after sorting, is shown in Figure 6.

The calculation of the Fundamental Sampling Error for each category considering every sampling step of the methodology was conducted using Equation 1 and ECHANT, a software based on TOS dedicated to the calculation of FSE.[12]

Figure 7 shows the results in terms of relative errors at 95 % confidence level, as well as the unit masses used for the FSE calculations for each heteroclite objects category (in red). The relative FSE associated with the proportion of the heteroclite objects and of the rest (representing about 81.4 %) is also calculated (in green). According to the dry method, the part of the sample, without heteroclite objects fraction, was quartered before screening and sorting.

Figure 8 shows the results for each >100 mm fraction category (in red) and the <20 mm fine element category (in blue). The mass of the batch taken into account here for the calculation (designated as secondary batch) is no longer equal to infinity, but is equal to about 234 kg, the mass of the initial sample *without* heteroclite objects. The mass of sorted sample (58.5 kg) corresponds to the mass obtained after quartering of the secondary batch. For each category, the resulting FSE is not the total FSE, but only these one generated by the quartering step.

Here again, the FSE generated by the sample screening is also calculated for both fractions >100 mm and 20–100 mm (in green).

Figure 9 shows the results for each 20–100 mm fraction category (in red). The mass of the batch taken into account for the calculation (designed as the final batch) is equal to about 28 kg, corresponding to the total mass of the 20–100 mm fraction after the previous step. The mass of sample sorted (5 kg) corresponds to the mass recommended by the MODECOM protocol for this fraction.

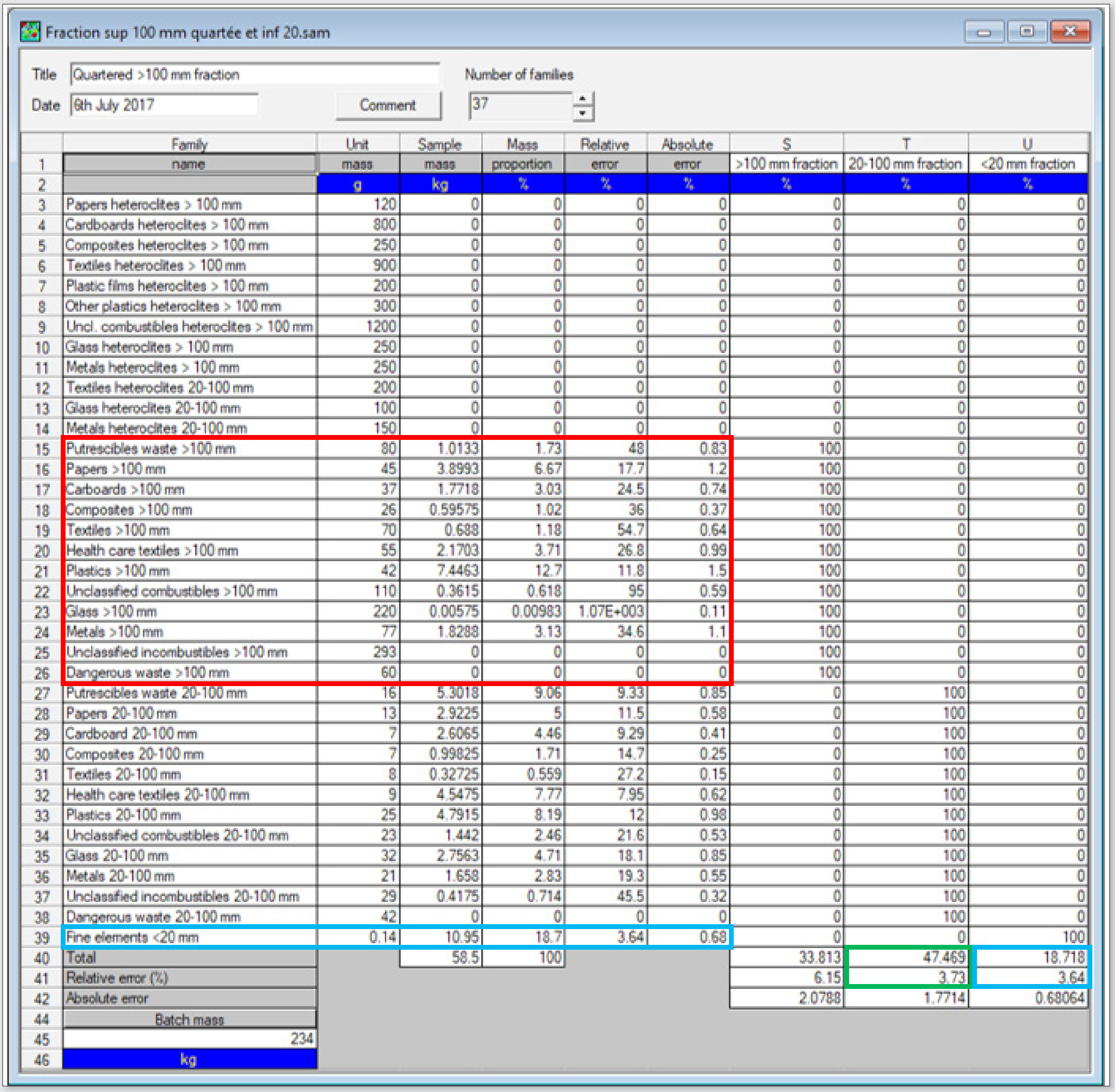
This step is the last one in the dry sorting approach when the <20 mm fraction is not sorted into categories. For each 20–100 mm category, the resulting FSE is not the total FSE, but only the one generated by the final step.

From the above results, it is now possible to calculate the *total* Fundamental Sampling Error for each of the categories by considering the variance of the FSE generated at each sampling (or quartering) step following appropriate error propagation rules.[9] The resulting FSE for the considered sample is detailed in Table 2 and error bars associated with the proportions in Figure 10.

According to these results, it can be seen that the Fundamental Sampling Error is not the same across all categories, in fact it varies significantly. For example, based on the considered raw sample of 512 kg, corresponding to a dry mass of 287 kg, "Glass >100 mm" represents 0.5 % associated with a relative FSE equal to 1073 %. In this case, the mass of the category "Glass >100 mm" is only 23 g in the sorted sample, while the mean unit mass taken into account for this category is 220 g; this is typically the case of a *nugget effect* and, strictly speaking, Pierre Gy's FSE formula cannot, and should not, be applied in such cases.

On the other hand, "Putrescible waste 20–100 mm" represents 7.4 % associated with a relative FSE equal to only 22 %.

In other words, this case highlights that a sample mass of 500 kg is, in general, not



Title: Quartered >100 mm fraction

Date: 6th July 2017

Number of families: 37

	Family	Unit	Sample	Mass	Relative	Absolute	S	T	U
1	name	mass	mass	proportion	error	error	>100 mm fraction	20-100 mm fraction	<20 mm fraction
2		g	kg	%	%	%	%	%	%
3	Papers heteroclites > 100 mm	120	0	0	0	0	0	0	0
4	Cardboards heteroclites > 100 mm	800	0	0	0	0	0	0	0
5	Composites heteroclites > 100 mm	250	0	0	0	0	0	0	0
6	Textiles heteroclites > 100 mm	900	0	0	0	0	0	0	0
7	Plastic films heteroclites > 100 mm	200	0	0	0	0	0	0	0
8	Other plastics heteroclites > 100 mm	300	0	0	0	0	0	0	0
9	Uncl. combustibles heteroclites > 100 mm	1200	0	0	0	0	0	0	0
10	Glass heteroclites > 100 mm	250	0	0	0	0	0	0	0
11	Metals heteroclites > 100 mm	250	0	0	0	0	0	0	0
12	Textiles heteroclites 20-100 mm	200	0	0	0	0	0	0	0
13	Glass heteroclites 20-100 mm	100	0	0	0	0	0	0	0
14	Metals heteroclites 20-100 mm	150	0	0	0	0	0	0	0
15	Putrescibles waste >100 mm	80	1.0133	1.73	48	0.83	100	0	0
16	Papers >100 mm	45	3.8993	6.67	17.7	1.2	100	0	0
17	Carboards >100 mm	37	1.7718	3.03	24.5	0.74	100	0	0
18	Composites >100 mm	26	0.59575	1.02	36	0.37	100	0	0
19	Textiles >100 mm	70	0.688	1.18	54.7	0.64	100	0	0
20	Health care textiles >100 mm	55	2.1703	3.71	26.8	0.99	100	0	0
21	Plastics >100 mm	42	7.4463	12.7	11.8	1.5	100	0	0
22	Unclassified combustibles >100 mm	110	0.3615	0.618	95	0.59	100	0	0
23	Glass >100 mm	220	0.00575	0.00983	1.07E+003	0.11	100	0	0
24	Metals >100 mm	77	1.8288	3.13	34.6	1.1	100	0	0
25	Unclassified incombustibles >100 mm	293	0	0	0	0	100	0	0
26	Dangerous waste >100 mm	60	0	0	0	0	100	0	0
27	Putrescibles waste 20-100 mm	16	5.3018	9.06	9.33	0.85	0	100	0
28	Papers 20-100 mm	13	2.9225	5	11.5	0.58	0	100	0
29	Cardboard 20-100 mm	7	2.6065	4.46	9.29	0.41	0	100	0
30	Composites 20-100 mm	7	0.99825	1.71	14.7	0.25	0	100	0
31	Textiles 20-100 mm	8	0.32725	0.559	27.2	0.15	0	100	0
32	Health care textiles 20-100 mm	9	4.5475	7.77	7.95	0.62	0	100	0
33	Plastics 20-100 mm	25	4.7915	8.19	12	0.98	0	100	0
34	Unclassified combustibles 20-100 mm	23	1.442	2.46	21.6	0.53	0	100	0
35	Glass 20-100 mm	32	2.7563	4.71	18.1	0.85	0	100	0
36	Metals 20-100 mm	21	1.658	2.83	19.3	0.55	0	100	0
37	Unclassified incombustibles 20-100 mm	29	0.4175	0.714	45.5	0.32	0	100	0
38	Dangerous waste 20-100 mm	42	0	0	0	0	0	100	0
39	Fine elements <20 mm	0.14	10.95	18.7	3.64	0.68	0	0	100
40	Total		58.5	100			33.813	47.469	18.718
41	Relative error (%)						6.15	3.73	3.64
42	Absolute error						2.0788	1.7714	0.68064
44	Batch mass								
45	234								
46	kg								

Figure 8. Relative fundamental sampling errors (FSE) at 95 % confidence per category of >100 mm fraction and <20 mm fine elements after the quartering step.

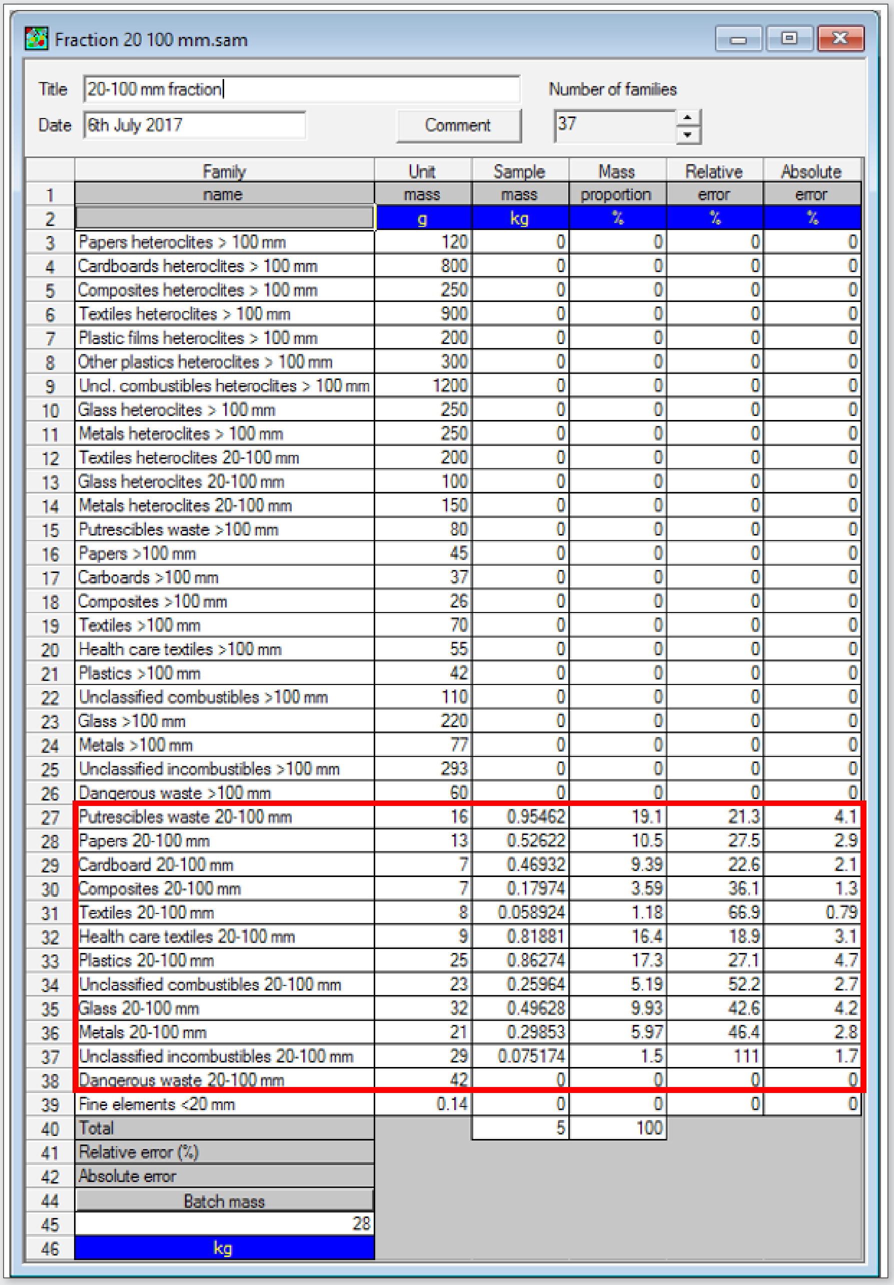
sufficient to have a good accuracy regarding the proportion of "Glass >100 mm". It is important to note that the mass of 500 kg recommended in MODECOM corresponds to a *compromise* between the time required for sorting, the associated cost and the accuracy of categories corresponding to the materials which are potentially *recyclable* when the methodology has been developed (this means mainly plastic-, metals- and cardboard-packaging, as well as papers).

Conclusions

The example presented shows that the Theory of Sampling can fully be applied to household waste. In France, the composition of MSW is determined using the MODECOM protocol from a stipulated 500 kg composite sample sorted into categories/sub-categories. Municipal solid waste is a highly heterogeneous material, so the composition resulting from sorting is associated with a total measurement error, for which the sampling error is the main component.

It is possible to calculate the Fundamental Sampling Error from data available in the literature. However, in the case of MSW, the mean unit mass for each category/sub-category is a critical parameter which can be difficult to determine experimentally, as this is time-consuming and often also expensive.

On a limited time-scale, the constituents of MSW are relatively stable. It is, therefore, possible to use unit masses coming from a database built up from large-scale



Fraction 20 100 mm.sam

Title 20-100 mm fraction

Date 6th July 2017

Number of families 37

	Family	Unit	Sample	Mass	Relative	Absolute
1	name	mass	mass	proportion	error	error
2		g	kg	%	%	%
3	Papers heteroclites > 100 mm	120	0	0	0	0
4	Cardboards heteroclites > 100 mm	800	0	0	0	0
5	Composites heteroclites > 100 mm	250	0	0	0	0
6	Textiles heteroclites > 100 mm	900	0	0	0	0
7	Plastic films heteroclites > 100 mm	200	0	0	0	0
8	Other plastics heteroclites > 100 mm	300	0	0	0	0
9	Uncl. combustibles heteroclites > 100 mm	1200	0	0	0	0
10	Glass heteroclites > 100 mm	250	0	0	0	0
11	Metals heteroclites > 100 mm	250	0	0	0	0
12	Textiles heteroclites 20-100 mm	200	0	0	0	0
13	Glass heteroclites 20-100 mm	100	0	0	0	0
14	Metals heteroclites 20-100 mm	150	0	0	0	0
15	Putrescibles waste >100 mm	80	0	0	0	0
16	Papers >100 mm	45	0	0	0	0
17	Carboards >100 mm	37	0	0	0	0
18	Composites >100 mm	26	0	0	0	0
19	Textiles >100 mm	70	0	0	0	0
20	Health care textiles >100 mm	55	0	0	0	0
21	Plastics >100 mm	42	0	0	0	0
22	Unclassified combustibles >100 mm	110	0	0	0	0
23	Glass >100 mm	220	0	0	0	0
24	Metals >100 mm	77	0	0	0	0
25	Unclassified incombustibles >100 mm	293	0	0	0	0
26	Dangerous waste >100 mm	60	0	0	0	0
27	Putrescibles waste 20-100 mm	16	0.95462	19.1	21.3	4.1
28	Papers 20-100 mm	13	0.52622	10.5	27.5	2.9
29	Cardboard 20-100 mm	7	0.46932	9.39	22.6	2.1
30	Composites 20-100 mm	7	0.17974	3.59	36.1	1.3
31	Textiles 20-100 mm	8	0.058924	1.18	66.9	0.79
32	Health care textiles 20-100 mm	9	0.81881	16.4	18.9	3.1
33	Plastics 20-100 mm	25	0.86274	17.3	27.1	4.7
34	Unclassified combustibles 20-100 mm	23	0.25964	5.19	52.2	2.7
35	Glass 20-100 mm	32	0.49628	9.93	42.6	4.2
36	Metals 20-100 mm	21	0.29853	5.97	46.4	2.8
37	Unclassified incombustibles 20-100 mm	29	0.075174	1.5	111	1.7
38	Dangerous waste 20-100 mm	42	0	0	0	0
39	Fine elements <20 mm	0.14	0	0	0	0
40	Total		5	100		
41	Relative error (%)					
42	Absolute error					
44	Batch mass					
45	28					
46	kg					

Figure 9. Relative fundamental sampling errors at 95 % confidence per category of the 20–100 mm fraction for a 5 kg sorted sample.

determination campaigns (national campaigns for example). Nevertheless, to take into account the variations related to local consumption behaviours, or changes in manufacturing processes for example, this database has to be updated regularly.

Considering the partitioning into categories/sub-categories *per size*, it can safely be assumed that the variability of the unit mass may be high within some categories/sub-categories. Thus, determinations of FSE from mean unit masses may easily lead to over- or under-estimations. Furthermore, while FSE gives a reliable estimate of sampling error in the ideal case, in the case of MSW, FSE represents only a part of the total sampling error, mainly because of their high constitution and distributional heterogeneity (CH and DH). But FSE is certainly the largest component.

Thus, the calculation of the Fundamental Sampling Error (FSE) associated with the composition of MSW following the approach presented in this paper, in the author's opinion represents a significant step forward regarding awareness of the significant heterogeneity of this type of material. This article presented a systematic procedure to estimate the specific FSE across the spectrum of standard categories following MODECOM.

References

1. K.C.R. Drudi, A.M.P. Neto, G. Martins, G.C. Antonio, J.T.C.L. Toneli, R. Drudi, C.H.S. Cenedese and L. Silva, "Physical analysis methods of municipal solid waste of Santo Andre", in *Waste: Solutions, Treatments and Opportunities*, Ed by Vilarinho, Castro and Russo). Taylor & Francis Group, London, pp. 73–78 (2015). doi: https://doi.org/10.1201/b18853-14
2. ADEME, "MODECOM. A method for characterisation of domestic waste", *Technical Guides and Manuals*. ADEME, Paris (1993).
3. AFNOR, *NF X30-413 – Household and Related Refuse – Constitution of a Sample of Household Waste Contained in a Waste Collection Vehicle*. AFNOR Publishing, Paris (2006).
4. AFNOR, *NF X30-408 – Household waste – Characterization method – Bulk product analysis*. AFNOR Publishing, Paris (2013).
5. AFNOR, *NF X30-466 - Household and related refuse –Characterisation method – Dry product analysis*. AFNOR Publishing, Paris (2013).
6. Ph. Wavrer and J. Villeneuve, "Determination of heavy metals in municipal waste streams", paper presented at First World Conference on Sampling and Blending, Esbjerg, 19–22 August (2003).
7. AFNOR, *NF X30-445 – Household and related refuse – Constitution of a sample of bulk household and related waste*. AFNOR Publishing, Paris (2006).
8. P. Gy, *Sampling of Particulate Materials, Theory and Practice*. Elsevier, Amsterdam (1979).
9. S. Brochot, "Application of sampling theory to optimal design of size distribution measurement procedures", in *Proceedings of the Sixth World Conference on Sampling and Blending 2013*, Ed by J. Beniscelli, J. Felipe Costa, O. Domínguez, S. Duggan, K.H. Esbensen, G. Lyman and B. Sanfurgo. Gecamine, Lima, pp. 129–140 (2013).
10. Ph. Wavrer, P. Botané and S. Brochot, "Sampling protocol design for characterization of plastics from small WEEE", in *Proceedings of the Sixth World Conference on Sampling and Blending 2013*, Ed by J. Beniscelli, J. Felipe Costa, O. Domínguez, S. Duggan, K.H. Esbensen, G. Lyman and B. Sanfurgo. Gecamine, Lima, pp. 143–152 (2013).
11. ADEME, *Adaptation of the MODECOM Method of Domestic Waste Characterization to Separate Collections – Determination of Sample Weights and Recommendations to*

Table 2. Total Fundamental Sampling Error budget (FSE).

Family name	**Mass proportion** *(dry %)*	**Total FSE variance**	**Total relative FSE** *(%)*	**Total absolute FSE** *(dry %)*
Putrescibles waste >100 mm	1.41	6.04E-02	48.2	0.68
Papers >100 mm	5.64	2.18E-01	91.5	5.16
Carboards >100 mm	5.21	1.16E-01	66.9	3.48
Composites >100 mm	1.04	4.74E-01	135.0	1.40
Textiles >100 mm	3.91	1.83E-01	83.8	3.28
Health care textiles >100 mm	3.02	1.91E-02	27.1	0.82
Plastics >100 mm	16.12	1.18E-01	67.3	10.86
Unclassified combustibles >100 mm	4.29	3.42E-01	114.6	4.92
Glass >100 mm	0.53	3.00E+01	1073.1	5.63
Metals >100 mm	3.28	1.55E-01	77.1	2.53
Unclassified incombustibles >100 mm	0	0	0	0
Dangerous waste >100 mm	0	0	0	0
Putrescibles waste 20-100 mm	7.38	1.26E-02	22.0	1.62
Papers 20-100 mm	4.07	2.05E-02	28.0	1.14
Cardboard 20-100 mm	3.63	1.41E-02	23.2	0.84
Composites 20-100 mm	1.39	3.47E-02	36.5	0.51
Textiles 20-100 mm	0.87	2.90E-01	105.5	0.92
Health care textiles 20-100 mm	6.33	1.01E-02	19.7	1.25
Plastics 20-100 mm	6.67	1.99E-02	27.6	1.84
Unclassified combustibles 20-100 mm	2.01	7.17E-02	52.5	1.05
Glass 20-100 mm	5.08	7.71E-02	54.4	2.76
Metals 20-100 mm	2.36	1.15E+00	210.3	4.96
Unclassified incombustibles 20-100 mm	0.58	3.22E-01	111.1	0.65
Dangerous waste 20-100 mm	0	0	0	0
Fine elements <20 mm	15.20	7.57E-04	5.4	0.82
	100			

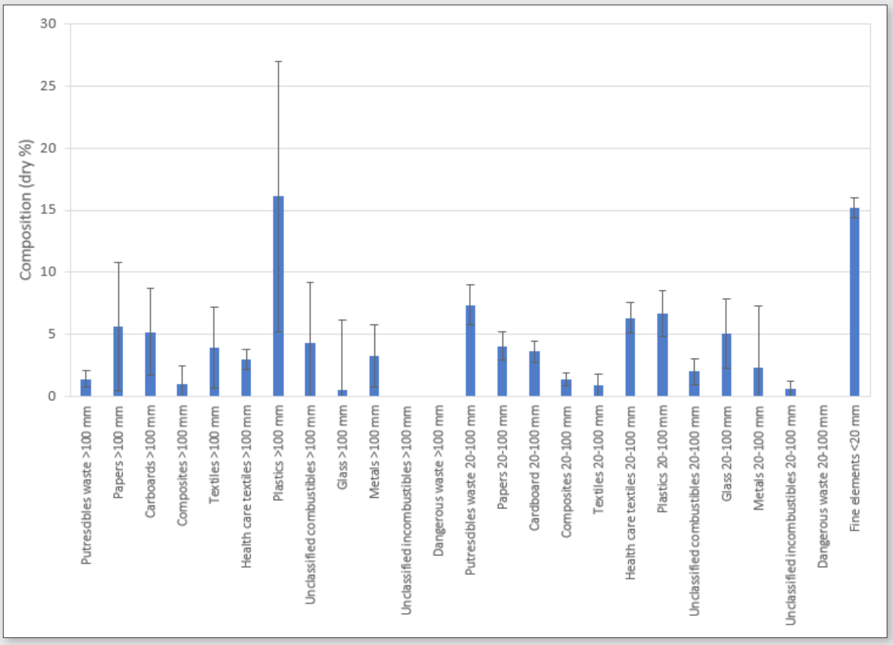


Figure 10. Total composition of the case study MSW and associated FSE (error bars).

Implement the Sample Collection. ANTEA report, contract No 9504163 (in French) (1996).

12. S. Brochot, *ECHANT: A Sampling Aid Tool*, paper presented at First World Conference on Sampling and Blending, Esbjerg, 19–22 August (2003).

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WCSB8 Conference Report

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On 9–11 May 2017, 206 delegates from around the world gathered in Perth for the 8th World Conference on Sampling and Blending (WCSB8), which on all accounts was a great success. The conference was the latest in the very successful conference series which commenced in Denmark in 2003 (WCSB1), followed by Australia in 2005 (WCSB2), Brazil in 2007 (WCSB3), South Africa in 2009 (WCSB4), Chile in 2011 (WCSB5), Peru in 2013 (WCSB6) and France in 2015 (WCSB7).

The conference was jointly hosted by The Australasian Institute of Mining and Metallurgy (The AusIMM) and the Australian Commonwealth Scientific and Industrial Research Organisation (CSIRO), with FLSmidth as a Platinum Sponsor, Thermo Fisher Scientific as a Gold Sponsor, IMP Innovative Solutions and Multotec as Silver Sponsors, and Herzog as the Sponsor of the Conference Proceedings. Additional support was provided by BHP Billiton (Technical Session), Process IQ (Name Tags) and Scantech (Note Pads and Pens).

The conference brought together most of all those globally involved in sampling and blending in the mineral, pharmaceutical, food, feed, agricultural, biomass, recycling and environmental monitoring sectors and industries. Delegates came from Australia, Belgium, Brazil, Canada, Chile, China, CIS, Czech Republic, Denmark, Finland, France, Germany, Italy, Mongolia, Netherlands, Norway, Papua New Guinea, Russia, South Africa, Turkey, UK and USA. Pierre Gy, founder of the Theory of Sampling (TOS) passed away in Bordeaux, France, in November 2015, so WCSB8 was dedicated to his memory and lifetime achievements in sampling commodities.

Figure 1. Professor Richard Minnitt with his freshly awarded Pierre Gy Sampling Gold Medal together with previous recipients (from left to right: Pentti Minkkinen, Francis Pitard, Kim H. Esbensen, Dominique François-Bongarçon, Ralph Holmes).

Figure 2. Pierre Gy Sampling Gold Medal.

Despite the wealth of knowledge available on correct sampling principles and practice, it is surprising how little attention and resources are sometimes devoted to extracting representative samples. Quite often everyone appears satisfied as long as *some* material is collected and delivered to the laboratory for analysis. Yet, unless the samples are representative, the whole measurement process is flawed at the outset and no amount of re-analysis can fix the fact that representativity is lost completely. As a direct result, companies often stand to lose millions of dollars in terms of poor investment decisions, wasted resources,

Figure 3. WCSB8 Organising Committee (from left to right: Simon Dominy, Andre Balt, Boyne Hohenstein, Antonia Riley, James Docherty, Claudia Paoletti, Darryl Stevens, Eliza Sanneman, Oscar Dominguez, Kim Esbensen, Ralph Holmes and Rodolfo Romañach).

Figure 4. Conference exhibition—visited by some of the absolute *illuminati* of sampling.

poor plant performance, poor product quality and sales income. Likewise, society often faces more than equivalent monetary losses, for the same technical reasons and also here resulting in poor decisions, but in addition serious, sometimes even fatal, inadequacy and irresponsibility regarding public health risks, food/feed quality deterioration, environmental pollution and resource over-consumption. Even though such consequences of inferior sampling are often hidden from direct observation, in sum they are of even greater economic significance. Sampling, therefore, needs to be given the attention it deserves by ensuring that the samples extracted are representative and that meaningful decisions can be made based on their analyses.

A key objective of the conference series is to bring to centre stage the importance of collecting representative samples at the outset ("Get your sampling right from the start"), and this objective was further reinforced by WCSB8. After a "Welcome to Country" by the Reverend Sealin Garlett on behalf of the traditional owners of the land on which the conference was being held, the conference was officially opened by Pamela Naidoo-Ameglio, a Director of The AusIMM. This was followed by the first of the seven Keynote presentations by a selection of highly regarded international sampling experts who shared their expertise and views on various aspects of sampling and blending over the three-day conference as follows:

Dr Francis Pitard: Integration of Visman and Ingamells' works into the Theory of Sampling—a necessity to expand possibilities in sampling practices

Dr Isobel Clark: Underground bulk sampling, uniform conditioning and conditional simulation—unrealistic expectations?

Dr Rodolfo Romañach: Theory of Sampling—from missing link to key enabler for process analytical technology

Dr Anita Parbhakar-Fox: Sampling and blending in environmental campaigns—current practice and future opportunities

Kathy Ehrig: Sampling the supergiant Olympic Dam iron-oxide Cu-U-Au-Ag deposit, South Australia

Dr Claudia Paoletti: Assessing and monitoring food and feed safety—a 15 year quest for proper sampling

Dr Kim Esbensen: Theory of Sampling—what's next?

The balance of the conference technical programme comprised more than 40 contributed papers covering a broad range of important and fascinating areas, including sampling theory, geostatistics, drill and blasthole sampling, quality control, metallurgical accounting, environmental sampling and monitoring, sampling of agricultural and biomass products, process analytical technology, and new developments and equipment.

There was also a discussion forum on the second day to provide delegates with the opportunity to seek answers to difficult sampling problems from a panel of international sampling experts. This forum

Table 1. The inaugural positions in the IPGSA.

Chair	Ralph Holmes	
Vice-Chair	Simon Dominy	
***TOS Forum* Editor**	Kim H. Esbensen	
Members	Ana Chieregati	Karin Engstrom
	Claudia Paoletti	Rodoldo Romanach
	Oscar Dominquez	Philippe Davin
	Elke Thisted	Trevor Bruce
Advisory Committee	Francis Pitard	Dominique Francois-Bongarcon
	Dick Minnitt	Pentti Minkkinen
	Geoff Lyman	

Figure 5. Conference exhibition—getting up to speed at an early moment.

was also used by the WCSB8 Chair, Dr Ralph Holmes, to preside over the launch the International Pierre Gy Sampling Association (IPGSA), the purpose of which is to oversee various formal activities of the international sampling community and represent the views of this community as appropriate from time to time, including awarding a country the right to host a World Conference on Sampling and Blending. The proposed Constitution of IPGSA was published in *TOS Forum* Issue 7.

Nominations for IPGSA positions were called and the inaugural appointments were agreed to in forum (Table 1).

A highlight of the conference was the announcement, at the official conference dinner, of the winner of the 2017 Pierre Gy Sampling Gold Medal, which was awarded to Professor Richard Minnitt, WITS University in Johannesburg, South Africa, for "Excellence in Teaching and Application of the Theory of Sampling". Richard worked for Anglo American and JCI before joining WITS University to teach technical valuation and mineral economics, where he currently holds the JCI Chair of Mineral Resources and Reserves. Dick was considered an impressive recipient of the Gold Medal by all present. The following morning, Professor Minnitt presented the inaugural Pierre Gy Gold Medal Oration at the conference morning opening, where he presented a scholarly exposé of the background and historical development of Pierre Gy's Theory of Sampling (see elsewhere in this issue).

A number of additional awards were also presented at the official conference dinner, on this occasion for the best overall paper, the best paper by a young author (as the prime author) and the best oral presentation. The overall quality of the papers and presentations at the conference was excellent, so deciding on the winners was a difficult task for the selection committee, the final winners being as follows:

Best overall paper: "The sampling characteristics of grains contaminated by mycotoxins", by G.J. Lyman and S.A. Tittlemier.

Best paper by a young author: "Blasthole sampling (replicate and variographic experiments) in LKAB open pit iron ore mines—fit for purpose representativity?", by K. Engström and K.H. Esbensen.

Best presentation: "Theory of Sampling—from missing link to key enabler for process analytical technology", by R. Romañach.

The associated exhibition was quite large for a sampling and blending conference, and attracted 21 exhibitors, including Scantech International, Foraco Australia, Consep Pty Ltd, Bureau Veritas, Pilbara Standards Pty Ltd, Metzke, Rocklabs, Intertek, Bruker, IMP Group, Real Time Instruments, Multotec Process Equipment (Pty) Ltd, Process IQ, Iteca Socadei, FLSmidth Pty Limited, Autorun Technologies, Expedio, Thermo Scientific, PANalytical, Sodern and SMC Pneumatics. The coffee breaks and lunch were held in the exhibition area, so attention opportunities were maximised and there were great opportunities for networking.

A number of pre- and post-conference workshops were also held in association with WCSB8, which complemented the main technical programme and exhibition. These workshops were:

"Sampling Theory, Sampling Practices and their Economic Impact", by Dr Francis Pitard and Dr Dominique François-Bongarçon.

"A Practical Guide to Designing and Running Effective Sampling Programs", by John Graindorge (Snowden).

"Grade Control in Underground Gold Operations", by Dr Simon Dominy.

A post-conference tour of sampling equipment manufacture and laboratories in the Perth area was organised on the Friday following the conference. Delegates had the opportunity to visit and inspect FLSmidth's excellent facilities for manufacturing sampling equipment and the first class sample preparation and analysis laboratories of MinAnalytical and SGS.

Figure 6. WCSB8 Chair with WCSB9 bidding team from China (from left to right: Huachang Li, General Manager, BGRIMM MTC Technology, Shufang Tang, BGRIMM MTC Technology, Ralph Holmes, WCSB8 Chair, and Roy Xu, General Manager, Unismart Events Limited).

Figure 7. Post-conference sampling and laboratory tour.

In conclusion, thanks are due to the Organising Committee, The AusIMM Events and Publications Departments, authors, paper referees, sessions chairs, and all the Sponsors and Exhibitors, particularly FLSmidth as the Platinum Sponsor of the conference and Herzog as the Sponsor of the Conference Proceedings, for making WCSB8 such a success. Bids for hosting WCSB9 in 2019 were presented by South Africa and China during the forum session on Day 2. Both bids were attractive and were carefully considered by the newly established IPGSA, which by majority vote decided to award WCSB9 to China. Hence, we look forward to meeting again in 2019 in Beijing, China.

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WHAT is wrong with this sampler?

A photographic "drive-by shooting"

It is quite some time this column was featured last—not for want of suitable "items", but rather due to a too-busy schedule. Recently, however, the following item was brought to the attention of the Editor. The photographic documentation below is the result of a photographic "drive-by shooting" from a public road.

Much can be said about this accidental sighting. The positive aspect always comes first. This *could* very well be the most inexpensive, fully automated "sampling solution" on record; so a big A+ for these aspects ;-)

But this is not all, of course. This also *could be* the most *unlucky* amateur sampler design ever (but one can never be sure). As always, what is important here is **not** *where* the photos were taken, or *which* company is currently making use of this unfortunate sampler, but **only**: "WHAT is wrong with this sampler?" Please remember, this column is published exclusively for TOS educational purposes.

The Editor presented these photos to a series of international sampling experts, asking for immediate comments, which follow:

- "My heartfelt response would be unpublishable. This reminds me of a night at the Crown Casino—pure gambling."
- "Wheel of Fortune"—there could not be a more apt name for this contraption.
- "Fascinating... but is it a children's toy?"
- "I count at least three Incorrect Sampling Errors (ISE)—most impressive."
- "A thoroughly biased primary sampling, or rather 'specimenting'."
- "…and also: what about the sub-sampling of the primary material cone?"
- "As the consultant said to the client: what number do you want, pick a number any number you'd like."
- "This is one of the worst samplers I have seen. It's a joke, sadly."
- "The managers get a result, possible with high analytical precision, but they do not get accuracy."
- "This is yet another example showing the critical need for education on correct sampling."
- "This sampler performs every possible INCREMENT MATERIALISATION ERROR instead of proper sampling."

Q.E.D.

Figure 1. What caught the eye… (Photo: the Editor).

Figure 2. Upon closer inspection… The TOS-mind boggles… One is reminded of a Monty Python sketch, in which an erstwhile architect declares: "… passing by the *rotating knives*" (Photo: the Editor).

Figure 3. A-ha, the full picture—a two-step sampling solution. Sub-sampling of the primary "sample cone" is also needed (Photo: the Editor).

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The Pierre Gy Oration

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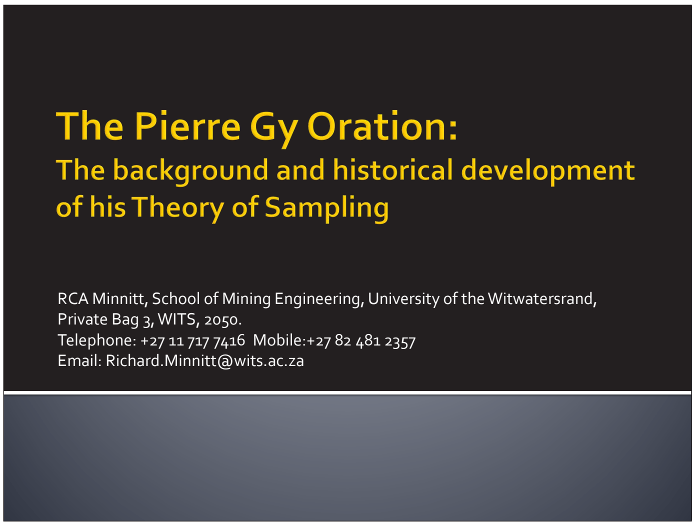
This is a graphic rendition of the inaugural “Pierre Gy Oration”, a new institution affiliated with the Pierre Gy Sampling Gold Medal (PGSGM). The honour of being a recipient of the PGSGM is, henceforward, associated with the obligation to give this distinctive speech as the lead-in to the morning session after the WCSB gala dinner at which the medal is awarded. Richard Minnitt was the first to fulfil this honour. His presentation turned out to be a veritable feast for the eyes, in fact it was so enticing that it was decided to present it here in its original PowerPoint format, in order to give readers the most vivid impression possible. Enjoy Dick’s comprehensive tribute to the founder of the Theory of Sampling.



The Pierre Gy Oration:

The background and historical development of his Theory of Sampling

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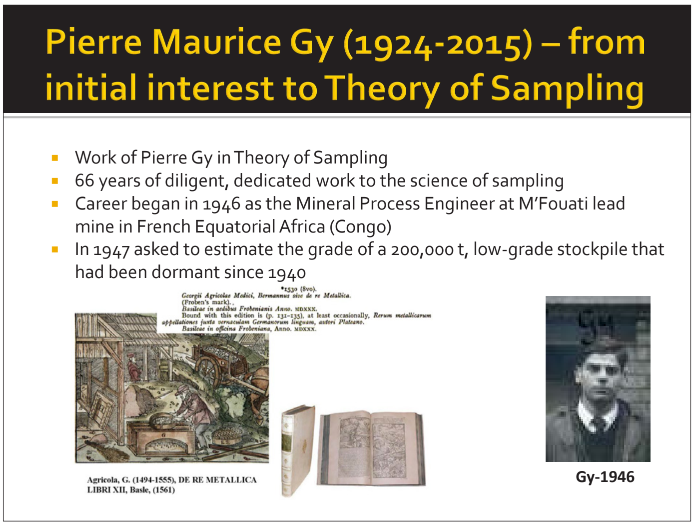


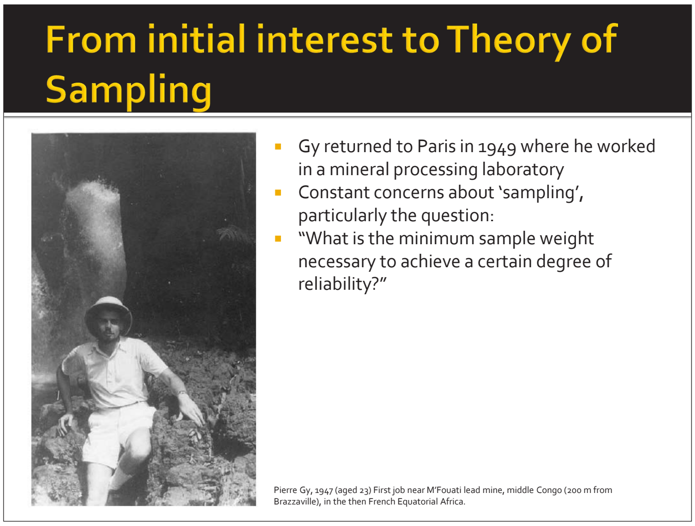
Pierre Maurice Gy (1924-2015) – from initial interest to Theory of Sampling

- Work of Pierre Gy in Theory of Sampling
- 66 years of diligent, dedicated work to the science of sampling
- Career began in 1946 as the Mineral Process Engineer at M’Fouati lead mine in French Equatorial Africa (Congo)
- In 1947 asked to estimate the grade of a 200,000 t, low-grade stockpile that had been dormant since 1940

*1530 (8vo).
Georgii Agricolae Medici, Bermannus sive de re Metallica.
(Froben’s mark).
Basileae in aedibus Frobenianis Anno. MDXXX.
Bound with this edition is (p. 131-135), at least occasionally, *Rerum metallicarum appellationes juxta vernaculam Germanorum linguam, autori Plateano.*
Basileae in officina Frobeniana, Anno. MDXXX.

Agricola, G. (1494-1555), DE RE METALLICA LIBRI XII, Basle, (1561)

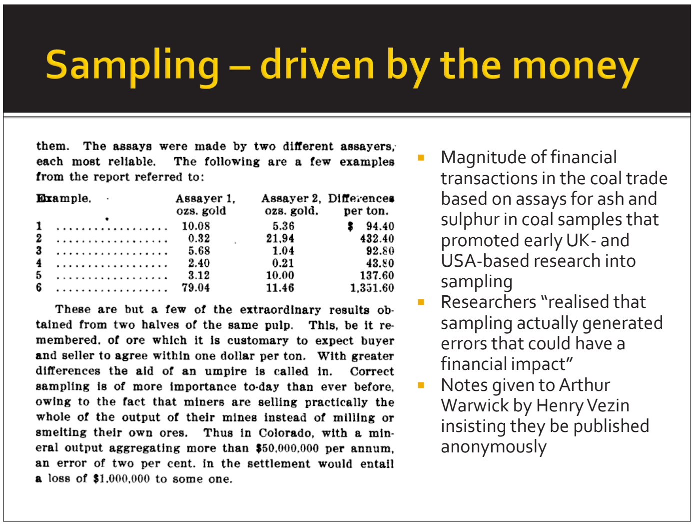
Gy-1946



From initial interest to Theory of Sampling

- Gy returned to Paris in 1949 where he worked in a mineral processing laboratory
- Constant concerns about ‘sampling’, particularly the question:
- “What is the minimum sample weight necessary to achieve a certain degree of reliability?”

Pierre Gy, 1947 (aged 23) First job near M’Fouati lead mine, middle Congo (200 m from Brazzaville), in the then French Equatorial Africa.



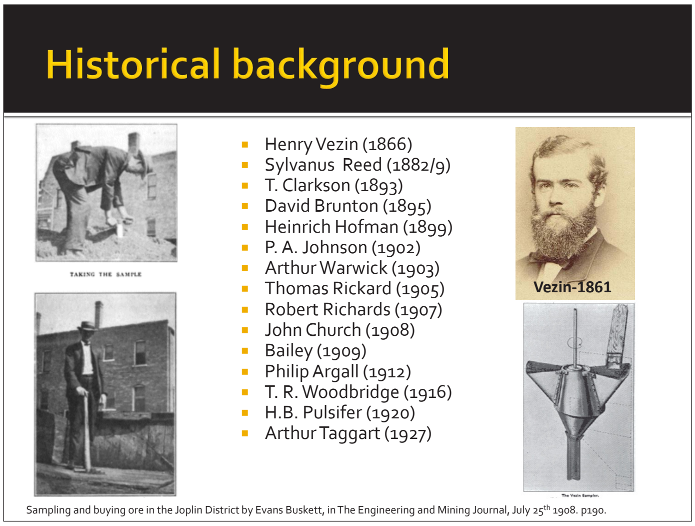
Sampling – driven by the money

them. The assays were made by two different assayers, each most reliable. The following are a few examples from the report referred to:

Example.	Assayer 1, ozs. gold	Assayer 2, ozs. gold.	Differences per ton.
1	10.08	5.36	$ 94.40
2	0.32	21,94	432.40
3	5.68	1.04	92.80
4	2.40	0.21	43.80
5	3.12	10.00	137.60
6	79.04	11.46	1,351.60

These are but a few of the extraordinary results obtained from two halves of the same pulp. This, be it remembered, of ore which it is customary to expect buyer and seller to agree within one dollar per ton. With greater differences the aid of an umpire is called in. Correct sampling is of more importance to-day than ever before, owing to the fact that miners are selling practically the whole of the output of their mines instead of milling or smelting their own ores. Thus in Colorado, with a mineral output aggregating more than $50,000,000 per annum, an error of two per cent. in the settlement would entail a loss of $1,000,000 to some one.

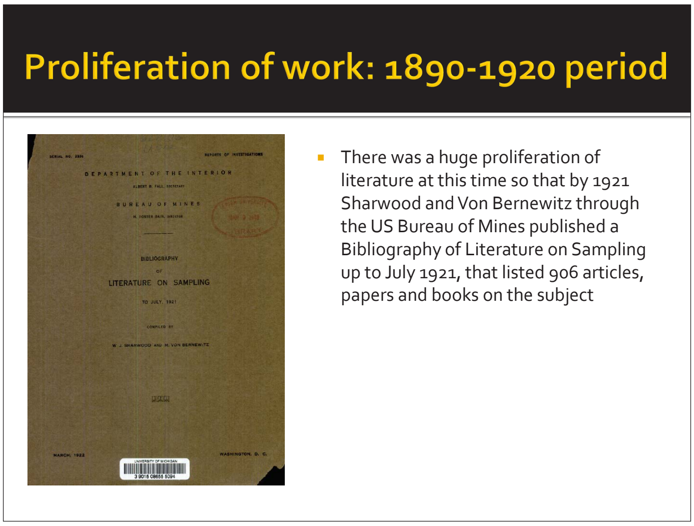
- Magnitude of financial transactions in the coal trade based on assays for ash and sulphur in coal samples that promoted early UK- and USA-based research into sampling
- Researchers “realised that sampling actually generated errors that could have a financial impact”
- Notes given to Arthur Warwick by Henry Vezin insisting they be published anonymously



Historical background

- Henry Vezin (1866)
- Sylvanus Reed (1882/9)
- T. Clarkson (1893)
- David Brunton (1895)
- Heinrich Hofman (1899)
- P. A. Johnson (1902)
- Arthur Warwick (1903)
- Thomas Rickard (1905)
- Robert Richards (1907)
- John Church (1908)
- Bailey (1909)
- Philip Argall (1912)
- T. R. Woodbridge (1916)
- H.B. Pulsifer (1920)
- Arthur Taggart (1927)

Sampling and buying ore in the Joplin District by Evans Buskett, in The Engineering and Mining Journal, July 25th 1908. p190.



Proliferation of work: 1890-1920 period

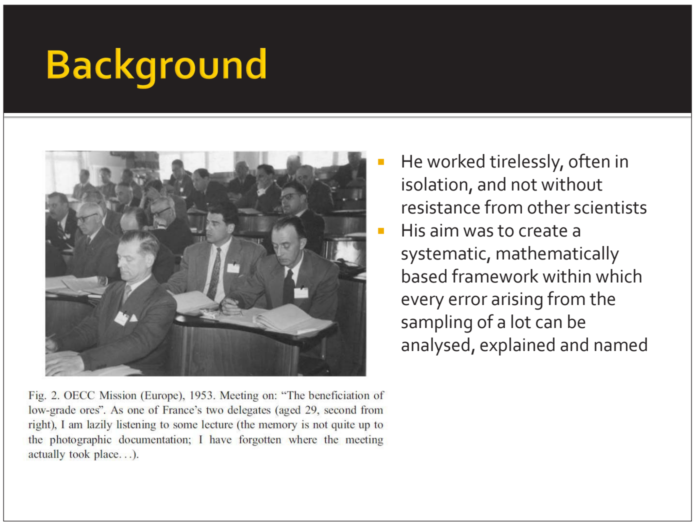
SERIAL NO. 2336 REPORTS OF INVESTIGATIONS

DEPARTMENT OF THE INTERIOR
ALBERT B. FALL, SECRETARY
BUREAU OF MINES
H. FOSTER BAIN, DIRECTOR

BIBLIOGRAPHY
OF
LITERATURE ON SAMPLING
TO JULY, 1921
COMPILED BY
W. J. SHARWOOD AND M. VON BERNEWITZ

MARCH, 1922 WASHINGTON, D. C.

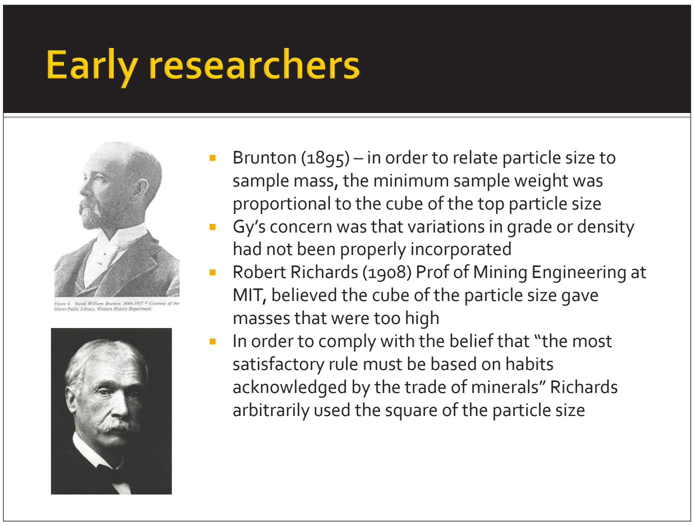
- There was a huge proliferation of literature at this time so that by 1921 Sharwood and Von Bernewitz through the US Bureau of Mines published a Bibliography of Literature on Sampling up to July 1921, that listed 906 articles, papers and books on the subject



Background

Fig. 2. OECC Mission (Europe), 1953. Meeting on: "The beneficiation of low-grade ores". As one of France's two delegates (aged 29, second from right), I am lazily listening to some lecture (the memory is not quite up to the photographic documentation; I have forgotten where the meeting actually took place. . .).

- He worked tirelessly, often in isolation, and not without resistance from other scientists
- His aim was to create a systematic, mathematically based framework within which every error arising from the sampling of a lot can be analysed, explained and named



Early researchers

Figure 3. David William Brunton 1849–1927.[10] Courtesy of the Denver Public Library, Western History Department.

- Brunton (1895) – in order to relate particle size to sample mass, the minimum sample weight was proportional to the cube of the top particle size
- Gy's concern was that variations in grade or density had not been properly incorporated
- Robert Richards (1908) Prof of Mining Engineering at MIT, believed the cube of the particle size gave masses that were too high
- In order to comply with the belief that "the most satisfactory rule must be based on habits acknowledged by the trade of minerals" Richards arbitrarily used the square of the particle size

Pierre Gy's early publications

- Gy's own mathematical model related variance of the sampling error to M_L, M_S, and physical properties of the material being sampled
- "The minimum sample mass for an acceptable sampling variance"
- No support in terms of time or resources for this research
- He devised a formula and the basic tenets of Theory of Sampling in two internal, unpublished notes for his company *Ste Minerais et Metaux*, entitled: "A formula for the minimum sample mass" and "Minimum sample mass required to represent a batch of ore" as early as 1950.

- P Gy (1953) "Erreur commise dans le prélèvement d'un échantillon sur un lot de minerai" Congres des Lavriesdes Mines Metalliques, Paris, September1953
- P Gy(1954) "Erreur commise dans le prélèvement d'un échantillon sur un lot de minerai" Congrès des laveries des mines métalliques françaises, Ecole des Mines de Paris Revue de l'IndustrieMinérale36 311 –345

The Formula Established

- Progression in Gy's 1950's logic is fascinating
- He identified physically well-defined parameters:
  - N_L the number of fragments in the lot
  - N_S the number of fragments in the sample
  - a_S grade of the sample
  - F_i the total number of fragments
  - M_i the average mass of fragments

- Strict algebraic relationships, simplified and approximated, for practical implementable formulae
- Mean and variance of population: "equally probable samples of N_S fragments"
- Needed good understanding in statistics – therefore earned a second PhD

Gy's equations

- Heterogeneity - most important concept, "lies at the root of all sampling errors"

$$h_i = \frac{(a_i - a_L)}{a_L} \times \frac{M_i}{M_i^*}$$

  - h_i - constitutional heterogeneity carried by one fragment F_i in the lot L
  - M^*_i - average mass of all individual fragments F_i
  - h_i – contribution to heterogeneity carried by each fragment in the lot
- Which when converted to an average variance h_i^2/N_L for the lot and divided by the number of fragments in the sample N_S given an appropriate statistical weight
- Approximate measure of variance of Total Sampling Error, $^2{}_{TSE}$

$$\sigma^2_{TSE} = \left(\frac{1}{N_S} - \frac{1}{N_L}\right) \cdot \frac{\sum_{i=1}^{N_L} h_i^2}{N_L}$$

The Formula

- Trials-and-errors Gy tested many simplifications and approximations
- Arrived at the well-known general equation

$$\sigma^2_{FSE} =\sim \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \cdot cfg\ell d^3$$
$$=\sim \frac{cfg\ell d^3}{M_S} \quad when\ M_S << M_L$$

- Referred to as "Gy's formula" , but he preferred 'The Formula'
- First presented in English to the Society of Mining Engineers of the American Institute of Mining Engineers (SME of AIME) in 1957
- Only in 1965 that his research was presented in London at a meeting of the Institution of Mining and Metallurgy (IMM).

Parameters for the Formula

- c - mineralogical composition factor; takes average grade and density of all components into account specific gravity (g/cm^3)
- c = /a_S -increases as average grade decreases
- ℓ - dimensionless liberation factor defined by Francois-Bongarcon as
- $\ell = (d_\ell/d_N)^{0.5}$ varies betwen 0 (liberated) to 1 (non-liberated)
- f - dimensionless particle shape factor, 0.5
- g - dimensionless size range factor, 0.25
- d - top particle size (cm) passing 95% of material

Probabilistic sampling model

- A highly simplified version of the probabilistic model is shown below:

$$\sigma^2_{FSE} = \frac{1-P}{PM_L} \cdot CH_L$$
$$= \left[\frac{1}{M_S} - \frac{1}{M_L}\right] HI_L$$

Where the Heterogeneity Invariant:
$HI_L = cfg\ell d^3$, and hence

$$\sigma^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] \cdot cfg\ell d^3$$

50th wedding anniversary with wife Sylvia, daughter Caroline and grandson Stanislas.

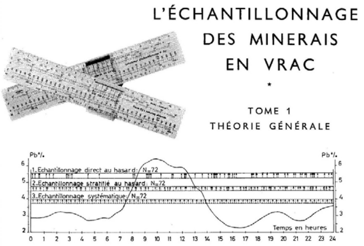
Extensions to the model

- His interest in sampling led to a first theoretical model for sampling of heterogeneous lots made up of particulate solids
- Then developed a generalised model for solids of animal and vegetable origin, types of domestic and industrial waste, liquids and gasses
- By this stage Gy recognised that the models had universal validity and that it was *scale* rather than physical state that differentiated between the range of applications.

Pierre Gy, extreme left, was 30 years old when this picture was taken in 1954.

Validating the Formula

- In validating his formula from the variance of sixteen equally split samples of pulverised lead ore, he found experimental errors larger than theoretical TSE:
- He therefore concluded that the Fundamental Sampling Error – is only one of several components
- Other components were GSE and sampling bias from incorrect use of riffle splitter
- Mid-1950's development of a circular cardboard sampling nomogram and later a sampling slide rule



Sampling of flowing streams

- Pierre Gy's 1960-1962 research into flowing streams of materials on conveyor belts and liquid launders
- Worked on velocity of cutters in cross-stream sampler, the width of the cutter opening and the shape of the cutter
- Gy recognised that increments extracted at constant intervals from a flowing stream are not independent from one another, there is some level of *auto-correlation* between time series sample data
- In 1962 Gy published work on *chronostatistics*, linear auto-correlation of time series data, borrowing the concepts of semi-variograms proposed by Matheron and later David
- Early '60's Gy chose writing and research of ToS over the comfort of Minerais et Metaux in Paris
- This began a grand forty-year period of theoretical research, consulting, lecturing and teaching regular courses

A period of opposition

- Serious opposition from members of the scientific communities 1967 French publication, "Sampling of Particulate Materials"
- ISO response was less than accepting of Gy's work
- His 1971 book entitled "Sampling of Particulate Materials, Volume 2" was soon followed by another book "The Theory and Practice of the Sampling of Particulate Materials" in 1975, but only a few hundred copies were ever sold
- Pierre Gy (2004a) tells of the difficulties he faced in 1978-1979 writing his first book in English, a translation of the 1975 text. The book, published in 1979, was followed by a second edition in 1982.
- Between the release of the Second Edition of the 1979 text and his latest book in French (1988), Gy developed a number of new applications of his theory including the computation of auxiliary functions of the variogram, the ideas underlying proportional sampling, and a theory of bed-blending

Sample selection – sampling errors

- Notion of "correct sampling" and its linkages to probabilistic sampling were first proposed by Gy in 1972.
- Concept in principle known by others (c.f. Vezin), Gy was the first to articulate sampling correctness "if, and only if, each and every fragment has the same statistical probability of being selected for the sample, as every other fragment".
- Gy built "the mathematical bridge between practical selecting conditions and theoretical sampling errors"
- Identified distinction between *a priori* conditions of sample selection, conditions we can do something about before taking the sample, and *posteriori* conditions of sampling selection, conditions we observe, but about which we can do very little after the fact
- Selection process: probabilistic <u>or</u> non-probabilistic, and even if probabilistic; it *could* be correct <u>or</u> incorrect.

Proportional Sampling

- Metallurgical balance reconciliation in a North African lead-zinc flotation plant
- "whatever comes in must ultimately come out, one way or another" with a single exception in 45 years, everything that ever came out was always <u>less</u> than what went
- Principal culprit for 2-3% deficit was calibration of conveyor belt scales
- Conveyor belt scales suffer from a structural lack of reliability, - converting electrical current to a measurement of ore tonnes
- Proportional sampler to metallurgical balances


1975,Suisse-Italie.
Au pied du Monte-Rosa
4600 m.

Pierre Gy in 1975, just turned 50

Proportional sampler

- Gy concluded that if the probability of selection P is a uniform distribution, then sampling is correct, and the mean of the sample mass M_S is a random variable equal to P times the mass of the lot M_L

$$m(M_S) = P \cdot M_L$$

- Accurate estimation of P means that M_S/P is an <u>unbiased</u> estimator of the mass of the lot, M_L
- All concentrates, tailings and feed streams, sampled according to the same selection probability make the proportionality factor constant.
- M_S/P is a more reliable estimator of mass of lot M_L, than any that can be obtained by weightometers and is the basis for idea of *proportional sampling*
- Gy defined a time sampling ratio and a mass sampling ratio

Derivation of Proportional Sampling

Explanation	Equation	Parameters
"time sampling ratio" of a lot, '	$\tau' = \frac{QT_i}{T_L}$	T_L = flow time of the lot L Q = No of increments, t=0 to t=T_L T_i = time to take one increment
"mass sampling ratio" of a lot,	$\tau = \frac{M_S}{M_L}$	M_S = mass of the sample S M_L = mass of the lot L
If sampling is correct	$\tau' = m(\tau)$	m( ) is the mean of
Rearranging	$\tau' = \frac{m(M_S)}{M_L}$	$M_L = \frac{m(M_S)}{\tau'}$

Sampling from all material streams is comparable, making the calculation of the material balance a simple task.

Bed blending

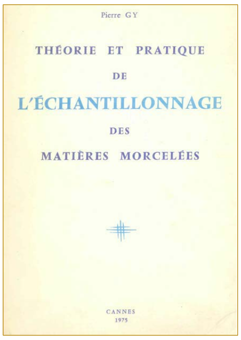
- Raw material feed to metallurgical furnace must be more-or-less uniform and homogenous
- Gy's bed blending studies began with input to cement kilns
- Costly damage to cement kilns can be avoided using uniform feeds
- Bed blending systems used to homogenise raw materials feed
- Good sampling equipment allowed major element analyses every few minutes
- Computerised assistance to calculate the average composition of the stockpiled kiln feed allowed the composition of the blending pile to be known with accuracy, providing an almost ideal feed to the kiln

Bed blending

- Gy found that bed blending theory was easily derived from existing sampling theory
- The manufacturer received an excellent explanation of how his equipment worked
- Gy's bed blending theory was in perfect agreement with practice and proved attractive to other cement producers
- Theory and practical aspects published in 1981 with a presentation to Canadian Institute of Mining and Metallurgy (CIM) in Montreal
- Theories that Gy published over the years have consistently proved to be correct, and were easily adapted to the science of bed blending.

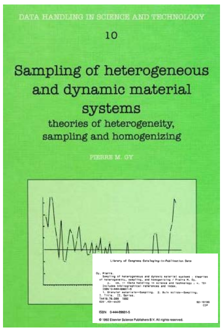
https://upload.wikimedia.org/wikipedia/commons/thumb/e/e9/KilnBZ.JPG/250px-KilnBZ.JPG

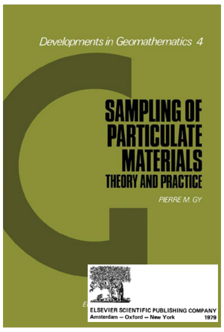
Pierre Gy's Publications

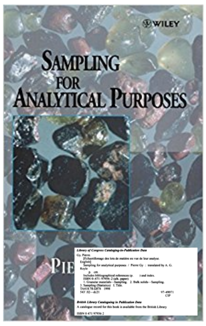


Pierre Gy with wife Sylvia at the WCSB1 banquet, Esbjerg, Denmark August 2003.

Pierre Gy's Publications





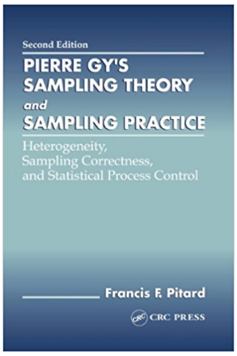


Francis F Pitard's English translation

- Industry standard has become the 1992 publication:
- "Pierre Gy's Sampling Theory and Sampling Practice,

Heterogeneity, Sampling Correctness and Statistical Process Control"

by Francis F Pitard



The legacy

- WCSB1 - Esbjerg, Denmark
- WCSB2 - Brisbane, Australia
- WCSB3 – Porto Alegre, Brazil
- WCSB4 – Cape Town, South Africa
- WCSB5 – Santiago, Chile
- WCSB6 – Lima, Peru
- WCSB7 – Bordeaux, France
- WCSB8 –Perth, Australia

- **TOS Forum,** Communication forum for the theory and practice of representative sampling (TOS) community

- **International Pierre Gy Sampling Association (IPGSA) (2017)**

doi: 10.1255/tosf.105

The 2010 Eyjafjellajökull volcanic ash sampling experiment: factors and conditions affecting field sampling variability

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[c]Specialist; Bioenergy and Thermal Power COWI Consulting, Denmark

In August 2010, a party of Icelandic explorers undertook a sampling experiment of the famous Eyjafjella volcanic eruption local ash falls. The possibility of having 10 two-person sampling groups with very different Theory of Sampling (TOS) competence and experience proved an opportunity too interesting to miss. The Eyafjellajökull field experiment constitutes a Replication Experiment performed on a lot of significant generic complexity and heterogeneity, illustrating primary sampling when there is absolutely no control over the original lot and its history. In such cases, the effects on the total sampling variance (i.e. the total Measurement Uncertainty) reflects a compound of factors that cannot be fully resolved even after a comprehensive experimental design. The ultimate confounding concerns sampling competence vs lot heterogeneity. The lessons learned have easy-to-interpret educational relevance for many other types of material lots with similar characteristics and heterogeneity in science, technology and industry. It was also a lot of fun.

Introduction

The Icelandic volcano Eyjafjallajökull erupted in April and May 2010, causing one the largest impacts in the global awareness of volcanic eruptions in modern society. Very few, if any, living in northern Europe missed out on the detrimental effect of several total or partial shutdowns of the European airspace. The eruption partly also caused massive delays and flight cancellations on a global scale.

There is an abundance of information readily available on the internet for this volcanic eruption, eyewitness accounts, video documentations, travel accounts, scores of scientific studies and publications ("just Google it"). Should the reader of this feature be inclined to delve a little deeper into the geology, three references serve as a convenient portal (even though the last is in the Danish language, it boasts a series of magnificent illustrations which alone make its very affordable price worthwhile).[1–3]

Among the many spectacular effects from this eruption which, paradoxically, by normal standards of Icelandic volcanism was classified as a "minor, average eruption", was heavy local ash falls in the areas surrounding the Myrdalsjökull glacier under which the Eyjafjalla volcanic fissure is located, especially along the eastern and northern flanks.

Some three months after the eruption ended, a party of 20 Icelandic field trippers (actually a double 60-year birthday party) visited the Myrdalsjökull's northern-most glacial tongue Gigajökjull and, amongst other things, conducted a sampling experiment on this local ash fall. There were three experienced sampling experts in the company (a geologist, a chemist and an engineer, all members of what today is the International Pierre Gy Sampling Association, IPGSA), who conducted a crash course for all other participants on the principles of representative sampling of near-surface sedimentary deposits, e.g. soil and strata-bound sediments, including ash falls, Figures 1–3.

Experimental design

The experiment was designed to illustrate the effects of the most important factors affecting field sampling variability: varying training and experience (Theory of Sampling, TOS), grab sampling vs composite sampling and material heterogeneity—*temporal* (it is known that the general composition of the erupting lavas, and hence of

Figure 1. The April/May 2010 Eyjafjallajökull volcanic eruption produced an unusual abundance of volcanic ash. The finest particles produced the by now (in)famous atmospheric ash cloud that caused havoc to North Atlantic air traffic and beyond. Locally severe ash falls were an equally spectacular feature. Figure 1 shows the development of ash fall deposits, which constitute the basis for a 2010 field sampling exercise with which to characterise field sampling variability. Photo: J. Helgason / Shutterstock.com

Figure 2. The August 2010 Eyjafjallajökull sampling variance experiment. Ten two-person field groups with widely varying sampling competences each acquired a "field sample" for chemical analysis of local ash fall deposits close to the distal, northern Myrdalsjökull glacier tongue (Gigajökull), Iceland. Analyses of all samples are presented in Table 2 and in Figures 6 and 7. Figure 3 details on-site training of field crews and show details of the ash fall sampling conditions (see text).

the ash produced, changed its composition significantly during the *ca* three-month eruption) as well as *spatial* compositional heterogeneity. Ash fall may undergo a significant grain size differentiation during atmospheric deposition,[1–3] Figure 1.

One of the most interesting factors, of general sampling interest, concerned the possible inter-personal effect made manifest by organising 10 two-person sampling parties with very different experience and initial competence regarding the TOS. After proper TOS initiation, Figure 3, each team decided individually *where* and *how* to sample inside a 500 × 500 m area underlain by *apparently* relatively uniform ash fall deposits, at least from its superficial expression, Figure 1. Ash fall sediments consist of microscopic glass fragments, resulting from violent explosive gas-fragmentation of molten rock magma in conjunction with rapid quenching. All groups had been instructed about the possible effect caused by *aeolean* stratification (*wind-blown particle size segregation*) likely to have been operating during the ash fall(s), but to an unknown degree, emphasising the critical need for a *complete* depth section through the recognisable top ash fall deposit stratum, Figure 4, though for volcanological reasons it is not necessarily to be expected that there would be very large compositional differences in different ash particle size fractions due to a single eruption ash flow, which was the target for this experiment.

More importantly, sampling the deeper pre-eruption surface deposits, viz. vegetated topsoil or earlier volcanic deposits, was to be avoided at all costs (in a mining sampling context this horizon is termed the *sub-drill*). Thus, the target was identified as the *latest* (top-most) ash fall unit. While this is a relatively simple target to identify in the field, at least for geologists, it nevertheless constitutes a potentially significantly heterogeneous lot material with a complex deposition history for which sampling may not necessarily be a simple affair. At any rate, in mid-August 2010 the ash fall was there, the party was there, TOS was in the air—in short this was an opportunity too interesting to miss.

Upon completed instruction and extensive group discussions, all groups agreed that a single-scoop sample, a "grab sample", could not be accepted as representative sampling in view of the apparent heterogeneity manifestations of the target material at both micro- and meso-scales (but, see also below, because of other "samples" of ash falls from the same eruption, these undoubtedly with a grab sampling provenance). A concord was quickly reached in the field that *composite sampling* was to be employed by all groups. There was a certain spread in the opinions on *how many increments* would be needed under the prevailing circumstances. This was left at the discretion of the individual sampling groups, for reasons to be clear below, and the results presented below thus represent between three and ten increments.

A general misconception was underlined: sampling is *not* a simple mass-handling process. While the ultimate analytical test portion requires only 0.4 g of ash, it was emphatically pointed out, actually by one of the least experienced participants: "Surely this is not as easy as to grab *any* 0.4 g with a scoop of the appropriate volume—the ash is visibly non-uniform at this scale level. How could I possibly demonstrate that my single-scoop 'sample' is representative?" This statement was sweet music to an experienced sampling expert's ears.

Figure 3. Field instruction on the principles of representative sampling (left). The inherent dangers of grab sampling were emphasised (right), as opposed to the virtues of composite sampling.

This field sampling exercise does not constitute a particularly easy task, either for newly initiated samplers or for the sampling specialist (or even for the geologist involved) because of the largely

Figure 4. Differential local surface erosion interfering with sampling of the last Eyjafjella ash fall. Taking this effect into account, the right-hand photograph depicts sampling a complete depth section through the latest May ash fall layer, guided by the underlying pre-eruption vegetation manifestations (equivalent to the "sub-drill"). A completely (ideal) sample for a two-dimensional lot, such as a sedimentary layer, constitutes an increment in the form of a cylindrical drill core. The degree to which successive increments of the form illustrated deviate from this cylindrical form will induce various Increment Delineation Errors (IDE); there may also be vestiges of Increment Extraction Errors (IEE) when the spoon is in the hands of inexperienced samplers, i.e. spillage.

unknown deposition details. Therefore, it constitutes a fair educational context from which hopefully to elucidate the factors that contribute to primary sampling variability with clarity.

For comparison, on the internet, the University of Iceland, Geological Institute published *contemporaneous* ash analyses spanning the entire three-month period March–May 2010, Table 1, with which the ten replicated primary samples from the August 2010 experiment can be compared. It was also possible to obtain commercially available "Eyjafjallajökull volcanic ash", claimed to be "representative" of this famous eruption, a feature that was well reflected in the price asked for every 20 g sachet commercially available to the Iceland tourist in the latter part of 2010. Two of three procured sachets of this origin were also included in the present experiment.

The details of the field experiment can be appreciated in Figures 2–4, which illustrate the general sampling scene, the sampling conditions, the target lot and its meso-scale material features. All sampling teams were issued with identical sampling tools.

Chemical analysis

The ten field samples obtained, Figure 2, were subsequently subjected to identical secondary sampling (mass reduction) and sample preparation procedures, which took place at the Geological Survey of Denmark and Greenland (GEUS), Copenhagen. This was carried out by the same, very experienced sampler (present author) in order not to introduce unnecessary, compounding sub-sampling errors at these stages. The subsequent chemical analysis was carried out in an ISO-9001 accredited laboratory, certified to analyse combustion fly ash for major and trace compositions. For the purpose of displaying the above effects on the total sampling variability, the major element compositions may perhaps have sufficed alone, but trace elements were specifically included as these are generally much more effective in reflecting compositional heterogeneity. All chemical analyses reported here are courtesy of Vattenfall DK.

Since all secondary and tertiary mass reduction, sample preparation and analysis were scrupulously identical, adhering to TOS' stringent demands, differences between the individual analytical results presented below will only reflect meso-scale ash heterogeneity *and/or* the varying degree of competence/experience *between* these 10 groups as regards the primary field sampling. But how to differentiate between these two factors?

Every analytical result has a provenance

The final analytical results will always reflect the inherent *confounding* between effects originating because of the heterogeneous nature of the lot material (FSE, GSE) and/or because of whether the sampling procedure used is correct, or not (bias-free, or not) along the entire field-to-analysis pathway (ISE).[4] Due to material heterogeneity, analytical results will be dependent on the *specific* sampling procedure employed. An alternative sampling procedure will in general lead to different numerical analytical results, to the degree that sampling procedures are either representative or not, and will also impact on the empirical sampling variance. Whether such differences will be large or small is never known in advance. In the present case, serious attempts were made to avoid the basic bias-generating grab sampling procedures at the primary sampling stage, Figure 3, as well as in all laboratory procedures.[a]

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[a] For reasons of keeping this article to a reasonable length, this is not the place also to present the basics of the Theory of Sampling (TOS), for which reason those seeking full explanations as to, for example, the so-called *correct sampling errors* (FSE, GSE), the complementary *incorrect*

Figure 5. Individual sampling groups in action.

Comparison, conditions and interpretations

As a means for comparison, the "official" rock and ash fall analyses published by the Earth Science Institute, Reykjavik University, will be used as benchmarks. As a group they in fact show a minor, but appreciable systematic difference to the field sampled samples in the present study, which is likely due to magma/lava compositional changes as the eruption progressed.[1,2]

But, more relevant to the present theme, there is no published information available as to the *methods* and *procedures* used for the sampling—*only* regarding analysis. There is nothing particularly unusual in this, however. The geological profession is, naturally, fiercely proud of their belief that: "… geologists *know* how to take a representative field sample", but this does not mean that TOS is irrelevant in the geological sciences. In fact, how to *assess* "representativity" based on non-complete sampling documentation, or a distinct lack thereof, often constitutes a moot issue in geology. All too often this issue is overlooked, or simply ignored, indeed it is often considered *irrelevant*: geologists train geologists, who train geologists, who train geologists… how to take a representative sample in the field. But *what if*… representativity is not a characteristic of the sample (of the analytical composition)? Such a thought is bordering on the heretical.

But, within TOS it is well known (this has been known for more than 60 years) that the adequacy and relevance of the analytical result *depends* on the specific sampling procedure used. It is fully possible to take a biased, non-representative sample (in TOS called a "specimen"), which is perhaps analysed with the ultimate precision, but the accuracy of which cannot be assessed. Within TOS it is well known that the qualifier "representative" can, and should, only be applied to the *sampling process*—not to the samples. It is not possible to ascertain the representativity status of a particular sample by its own characteristics—and this includes its analytical composition! Within this understanding, empirical sampling variability can arise both due to material heterogeneity *and/or* due to an inferior sampling process or even for other reasons (see footnote a above).

Staying with the traditional geological viewpoint, practice often devolves to a game of comparing total chemical analytical results at *face value*, i.e. as if analytical results always, universally are representative by *fiat* (when one does not know about sampling errors, there *are* no sampling errors). But the above argument explains why valid comparison is critically contingent upon full sampling documentation for all samples. Also: what is the status of a duly reported sampling procedure—representative, or not? It becomes clear that there is no such thing as a valid sampling comparison based on analytical results *alone*. This would be missing out completely regarding the provenance of the samples from which the analytical aliquot is but the last element.

Still, there is a perfectly feasible way out of this emerging dilemma: the TOS encompasses the complete set of ways and means needed in order to eliminate all bias-generating sampling errors (to ensure bias-free, accurate sampling) and further

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sampling errors (ISE), *sampling bias* a.o., are referred to the readily available introductory literature, e.g. References 5 and 6.

how to reduce the effect of the remaining imprecision errors, i.e. reducing the sampling variability. For the reader who has just received a scientific jolt and wishes to know more, referral is made to the entire sampling literature… a portal to which can be found in References 6 and 7.

A field Replication Experiment

Thus, the present field experiment presents a TOS feature at the educational forefront. particularly relevant as regards “Replication Sampling Variability (RSV)”.[8] The Eyjafjella experiment serves well to illustrate the framework of this approach: what is the total *empirical* variability displayed by ten “replicate primary samples” of the same lot material? It is vital that the lot in question is a realistic lot, not some form of a made-up reference lot. This is so because all lots have both a spatial as well as a compositional heterogeneity aspect (perhaps even a differentiating temporal origin as well).

Table 1. Official Eayafjallajökull lava and ash fall compositions. Chemical analysis* of rocks and ash from the Eyjafjöll 2010 eruptions, Níels Óskarsson; major oxides in wt %, trace elements in mg kg^{-1} (ppm).

Available from
http://earthice.hi.is/eyjafjallajokull_2010_chemical_composition

Indeed, by not paying the necessary attention to the representativity of the specific sampling procedure, it is actually possible to sample in a fashion, which can be characterised as “precisely wrong” (*precise* because of a low spread of ten analytical results, high precision, but *inaccurate*, *wrong* (because of non-representative sampling). In general, a physical “average sample”, a *composite sample* consisting of a number of representative increments, is always to be preferred over *any* single grab sample. In this context, the present experiment aimed for ten such superior primary composite samples in order to leave only the inherent ash heterogeneity as the main factor behind the observable sampling variability. But in order to stay within a realistic sampling context (often primary samplers are employed with considerable different training, competence and experience), the present setup between sampler groups is fully realistic. This experimental setup will further information analogous to “reproducibility” in an analytical chemistry setting.

An alternative version of the RE could, for example, call for ten replicate primary samples executed by the *same sampler* (of course, also using the exact same sampling procedure), harking to “repeatability” in analytical chemistry. It is important to be

Table 2. Ash fall compositions from the 2010 Eyjafjallajökull field sampling experiment. The six first entries are from Table 1.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	MnO	P_2O	Ba	Co	Cr	Cu	Ni	V	Zn
SRG-2b	57.98	14.87	9.75	5.50	2.30	1.79	5.01	1.80	0.24	0.53	447	27.00	25.0	27.0	15.0	86.0	144
SRG-5a	56.73	14.65	9.93	6.11	3.15	1.65	5.04	1.88	0.24	0.43	425	33.00	97.0	35.0	41.0	114	137
BO1510	59.26	14.55	9.29	4.35	2.50	1.89	5.46	1.38	0.26	0.48	489	24.00	43.0	28.0	36.0	66.0	192
HO7510	58.24	14.89	9.17	4.70	3.17	1.78	5.18	1.50	0.24	0.48	449	29.00	57.0	29.0	55.0	89.0	161
PAA8510	59.02	14.43	9.38	4.20	3.16	1.86	5.22	1.40	0.26	0.48	478	28.00	60.0	26.0	78.0	78.0	165
GSV165-3	59.59	14.36	8.55	4.15	3.52	2.02	5.24	1.33	0.22	0.40	467	30.00	63.0	29.0	70.0	91.0	188
C1	61.40	15.00	8.41	4.55	2.86	2.13	4.99	1.31	0.20	0.20	421	27.70	78.8	22.2	49.5	85.0	142
C2	61.00	14.90	8.50	4.54	3.02	2.15	4.93	1.32	0.20	0.20	412	28.30	79.7	22.2	52.8	85.1	140
C4	61.10	14.90	8.51	4.58	2.95	2.12	4.97	1.35	0.20	0.21	419	29.40	81.4	22.6	54.7	87.9	148
C5	60.70	14.80	8.57	4.57	3.17	2.12	4.84	1.30	0.20	0.20	415	30.20	89.3	22.5	63.4	87.7	145
C6	61.40	14.90	8.39	4.46	2.84	2.15	5.00	1.30	0.20	0.20	411	28.30	74.1	22.5	51.9	85.3	148
C7	60.70	14.90	8.63	4.64	3.02	2.09	4.92	1.40	0.20	0.22	404	31.30	75.8	24.9	53.0	99.6	147
C8	60.20	14.70	8.73	4.68	3.43	2.24	4.79	1.33	0.20	0.20	409	31.40	92.6	22.0	69.8	87.3	145
C9	61.00	14.90	8.46	4.56	2.90	2.13	4.96	1.32	0.20	0.21	426	30.00	84.3	22.9	56.8	88.9	150
C10	59.90	15.00	8.71	4.96	3.23	2.01	4.80	1.44	0.20	0.22	403	33.20	97.4	27.0	61.5	101	145
C3-1	61.40	15.00	8.30	4.34	2.71	2.19	5.04	1.28	0.19	0.20	417	28.10	71.2	22.2	53.0	84.3	143
C3-2	61.70	15.00	8.28	4.36	2.73	2.20	5.07	1.29	0.19	0.20	435	27.70	75.2	21.9	48.6	84.3	147
C3-3	61.50	14.90	8.27	4.34	2.72	2.19	5.08	1.28	0.19	0.20	417	27.30	72.0	21.2	50.1	83.0	145
C12-1	60.00	15.10	8.70	4.96	3.18	2.02	4.87	1.44	0.20	0.22	401	33.00	88.2	26.9	59.9	102	146
C12-2	60.00	15.10	8.68	4.95	3.18	2.04	4.87	1.42	0.20	0.21	400	32.80	90.5	27.3	60.8	102	146
C12-3	59.90	15.00	8.78	4.93	3.23	2.02	4.83	1.44	0.20	0.22	400	33.10	96.0	26.9	62.1	102	146

Chemical composition of top ash flow deposit from the Eyjafjöll 2010 eruption, 500 m north of terminal end of Gigajökull. Major oxides in w/w %, trace elements in ppm. Analysis: Vattenfall, DK (ISO-9001 certified ash characterisation laboratory). The two triple analytical replications described in the text are shown (C3-1, C3-2, C3-3 and C12-1, C12-2, C12-3..

Table 3. Analysis of variance (ANOVA) of field experiment data from Table 2. s^2 = variance, *F* = F-test, *Pr* = cumulative probability of the F-distribution at the value of *F*, *sr* = relative standard deviation (with respect to the mean). Explanation of subscripts: *ref* = commercial samples, *samp* = field samples, *a* = results from the two triplicate analyses and their pooled variance, *a* = analytical, *samp* = primary sampling. If *Pr(F)* is below 0.95 the sampling variance difference is not significantly different from zero at 95% confidence level.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	MnO	P_2O	Ba	Co	Cr	Cu	Ni	V	Zn
s^2_{ref}	1.10	0.049	0.23	0.64	0.22	0.015	0.026	0.055	0.00023	0.0021	545.0	9.10	572.7	10.0	541.4	254.3	499.5
s^2_{field}	0.264	0.0089	0.0196	0.0258	0.0453	0.0036	0.0075	0.0024	0.00001	0.00007	54.01	3.17	71.0	2.57	40.1	36.5	9.34
s^2_{a1}	0.0233	0.0033	0.00023	0.00013	0.0001	0.00003	0.00043	0.00003	0.0000	0.0000	108.0	0.16	4.48	0.26	5.00	0.56	4.00
s^2_{a2}	0.0033	0.0033	0.0028	0.00023	0.00083	0.00013	0.00053	0.00013	0.0000	0.00003	0.33	0.023	16.1	0.053	1.22	0	0
s^2_a	0.0133	0.0033	0.0015	0.00018	0.00047	0.00008	0.00048	0.00008	0.0000	0.00002	54.2	0.092	10.3	0.158	3.11	0.28	2.00
F	23.1	3.49	13.14	191.7	92.8	48.6	14.5	34.3	0	0	1.21	40.1	6.80	24.7	12.1	169.5	4.23
Pr(F)	0.994	0.821	0.987	1.000	1.0	0.99	0.991	0.997	0	0	0.455	0.998	0.961	0.992	0.987	1.000	0.924
s^2_{samp}	0.251	0.0056	0.018	0.026	0.045	0.0035	0.0070	0.0024	0.00001	0.00007	0	3.08	60.77	2.41	37.00	36.24	7.34
sr_a/%	0.19	0.38	0.46	0.29	0.73	0.43	0.44	0.67	0.00	1.96	1.79	1.00	3.90	1.63	3.16	0.57	0.97
sr_{samp}/%	0.82	0.50	1.58	3.49	7.03	2.79	1.70	3.64	1.59	3.58	0	5.89	9.45	6.72	10.7	6.75	1.87

fully aware, however, that this seeming parallel between the analytical process and the sampling-and-analysis process are, in fact, not comparable at all. This situation has been described in detail in Reference 9. The central issue pertains to what are the consequences of indiscriminate use of different samplers, with varying relevant competences. In an industrial context this is often permitted for "compelling" economic, efficiency or logistical reasons. In such a situation there is an impression that sampling is but simple "bulk materials handling", with only little focus on competence and education. Indeed, industrial primary sampling has often been subject to company *outsourcing* for extremely narrow-sighted budgetary reasons alone, while such scientific focus that may exist is typically more concerned with the type or brand of the equipment, the number of "replicates", or the *analytical method* s.s. All of which are indeed important factors, but which dwindle almost to insignificance when compared to the total effect of the many neglected sampling errors that go unnoticed without proper TOS insight.

Data analysis

The ten individual ash analytical results will be compared to the two available types of reference material results for the same target, Tables 1 and 2, and also graphically by use of multivariate data analytical *projections*, Figures 6 and 7. For the latter, PCA is employed. Suffice here to note that a principal component graphical plot allows easy visual comparison of the correlation between any number of variables; in the present case for a total of 17 variables (10 major element oxides and 7 trace elements), for any number of samples. PCA *projection* depicts the degree of *similarity* between samples by means of their Euclidian distances in so-called *score plots*, which is a reflection of their simultaneous compositional relationships to one-another (in PCA plots *closely* positioned samples have *closely* similar compositions for most, or all, of the variables involved). The sample disposition is "explained" by a complementary projection visualisation of the variable correlation relationships, in the corresponding *loading plots*. See Reference 4 for a full introduction to projection-based multivariate data analysis.

There is a critical aspect of the official data published in Table 1 that merits further discussion, and which well illustrates the themes taken up here. This concerns the use of "averages of duplicate analysis". What was *de facto* duplicated here: the analysis alone? Did this include sample preparation? Did it include sub-sampling? Or was it the primary field sampling? The latter options are highly unlikely in context, but there is no relevant information to be had. In a narrow analytical context, such issues are often not accorded further explanation, but this issue *must* be fully described, lest the users of the analytical results are unable to understand *from where* did the *averaged* analytical differences originate? What caused the analytical variability? It should be made perfectly clear that a comprehensive understanding of such ambivalent replication information is critical with respect to assessing the total sampling-and-analysis uncertainty, i.e. from *which level* in the full sampling-sub-sampling-sample-preparation-analysis pathway did the "duplication" (or "replication") take place? This issue is fully outlined, e.g., in References 4 and 8.

While the date of eruption, not necessarily the same date as that of sampling, plays an important role in understanding the overall evolution of the volcanic system over time and its intermittent eruptions, we will here leave this aspect to the geologists proper. Rather, focus is on the nature of the sampling methods (grab vs composite), which can be of equal influence on the total uncertainty budget, if not more so. By virtue of the experimental design, every effort was made here to sample only the singular, latest, top ash layer, so as to minimise such possible lava compositional differences. It is, therefore, the considered opinion that whatever differences in Table 2 owe their manifestations to the combination of ash fall heterogeneity × sampling representativity. It will not be possible to separate the influence from these two factors from another.[4,8] This is an important general condition for field sampling—identical for many other lot and material types.

From the analytical results in Table 2 it is possible to estimate the analytical variance and the primary sampling variance respectively. The latter reflecting the difference between sampling teams for all elements (except Al, Mn, P, Ba and Zn). The result of an Analysis of Variance (ANOVA) decomposition analysis of the data in Table 2 is given in Table 3.

The most obvious information in Table 3 is that the analytical precision is very good (all relative analytical standard deviations fall between 0.2 % and 4 %), attesting to excellent laboratory performance. By the same token, all ash-fall samples appear to be of a somewhat stable composition (relative sampling standard deviations range only 0.5–9.5 %). It is very illuminating to note that the primary sampling is responsible for the largest variance component in the experiment, because *average* sampling standard deviations compared to the analytical standard deviations in Table 3 amounts to *ca* 85 % across all chemical variables. Sampling process effects very nearly always dominate.

Thus, even if the ash fall in the designated experimental area after analysis turns out to be a much more uniform material than what was originally expected, there are clear, significant inter-sampler (inter-personal) variations. The largest contrasts relate to the elements present at the lowest concentration levels, e.g. MgO (present in an unusual low concentration for a major element) and the trace elements, fully as expected from geology and geochemistry.

Sampling case study: embedded replications

Several experimental design twists can be found behind the data in Table 2. There are three replicated analytical results pertaining to one primary field sample (C3), intending to capture the magnitude of the laboratory sub-sampling effects (samples Nos 3–5 in Table 2). Also, samples Nos 13–15 represent a similar three-fold analytical replication of the second commercially acquired “tourist sample”. Alas, the tourist samples come without *any* reported sampling details (no sampling date, no locality information, no sampling method specification). However, it is more than likely that they were acquired by *grab sampling*.

Both these triplications were made intending that the necessary aliquot subsampling operations to be included and evaluated together with the analysis (these sample triplications were not known to the laboratory). Thus, these triplicates were prepared for analysis by identical procedures to the ten field samples. These “triplicate analytical results” sets are compared in full in the multivariate data analysis below. The two “tourist samples” (C12 and C13/14/15, respectively) are also of interest because there would appear to be no reason to distrust their authenticity in view of their price in the Iceland souvenir kiosks! Tourists must, of course, be able to trust that they do indeed represent *bona fide* ash samples originating from the Eyafjallajökull explosive eruptive phase.

These commercially acquired samples therefore mimic a very often occurring general problem: “Samples” are required to be analysed, but little, or no, information exists as to their provenance and the specific sampling and sub-sampling employed. How often is an analytical laboratory not faced with this problem, and how often does the laboratory not simply *assume*, or is forced to assume, that the “samples” have been acquired by a representative sampling process. Or the analytical laboratory may opt to take the obvious, easiest way out: “Ours is not the responsibility to question the representativeness of samples received—our job to *analyse* the samples (only)”.

Below is a multivariate analysis of all the available chemical data relating to the sampling field experiment and these relevant comparisons. It is not possible to give a full presentation of all the necessary elements of multivariate data analysis, but the specific visualisation issues lend themselves to easy understanding. A complete introduction to multivariate data analysis, specifically PCA, can be found in Reference 5.

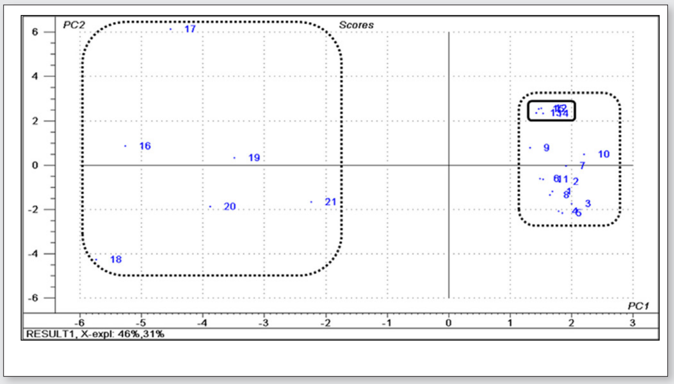

Figure 6A. PCA multivariate projection analysis (17 variables) for 11 field sample analyses (nos 1–11), four commercial analyses (nos 12–15) and six published analyses (nos 16–21).The major difference concerns the field/commercial samples (right) vs published samples (left). The first two principal components model (46 % + 31 %) = 79 % of the total data set variance.

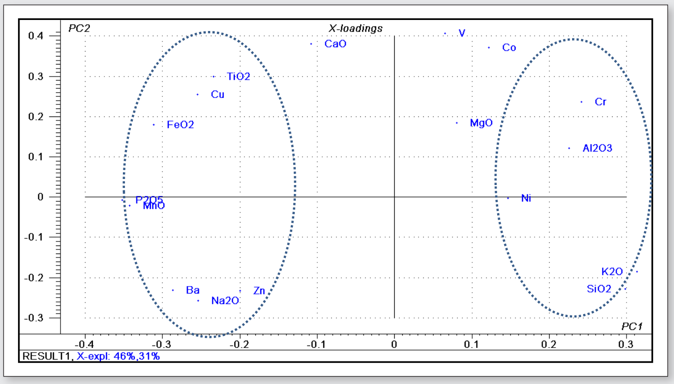

Figure 6B. Variable correlation pertaining to Figure 6A. From standard PCA interpretation, it is observed that the published analytical results are relatively enriched in the oxides of the elements in the left-hand side of this loading plot, while the field experiment samples are relatively enriched in the elements in the right-hand side. The first two principal components model (46 % + 31 %) = 79 % of the total data set variance.

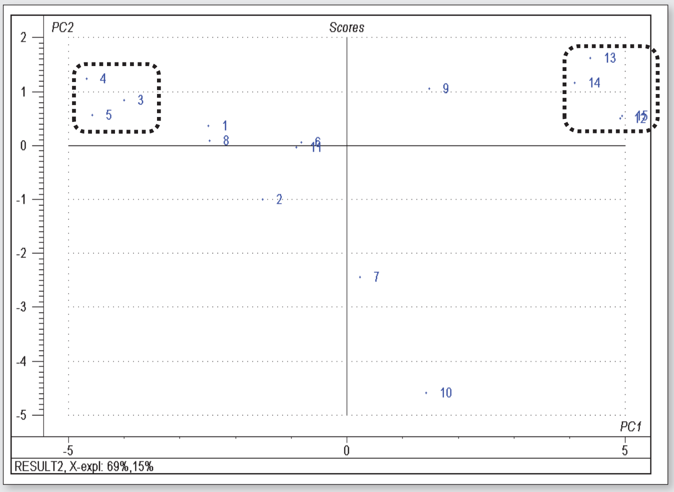

Figure 7A. Focused relationship between field samples and their commercial equivalents only. Commercial sample no. 12 was analysed once, while analyses 13–15 represent triplicate sub-sampling and analysis in the laboratory of the second commercial sample (score box right). Field sample 3 was similarly sub-sampled and analysed in triplicate (analyses 3, 4, 5) in the laboratory (square box right). These embedded replications illustrate the maximum laboratory aliquot handling-and-analysis error, with which to assess the sampling variability between all ten sampling group results. The first two principal components model (69 % + 15 %) = 84 % of the total data set variance.

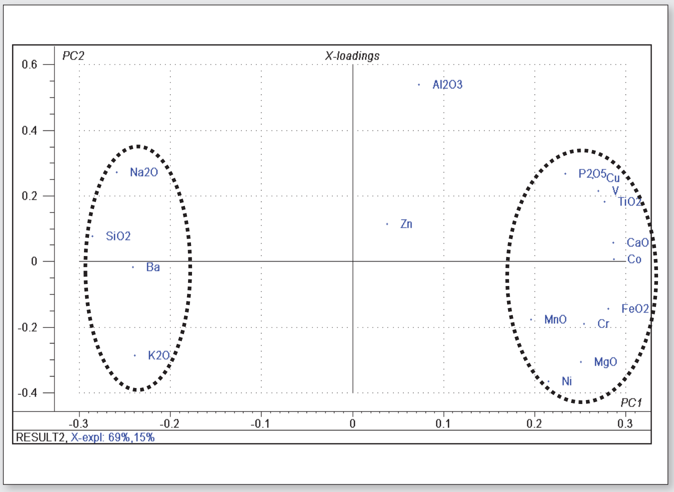

Figure 7B. Correlation relationships between variables for field and commercial samples in Figure 7A. The "silicic" (SiO_2, K_2O, Na_2O, Ba) vs "mafic" composition (MgO, CaO, FeO_2, Ni, Cr ...) differentiation is a well-known geochemical differentiation feature of basaltic volcanic lavas. The first two principal components model (69 % + 15 %) = 84 % of the total data set variance.

Technical note: for the data analytical cognoscenti

All variables in the principal component analysis (PCA) have been *auto-scaled*, i.e. *centred* (w.r.t. the average of all variables) and *normalised*, i.e. divided by 1/std. This facility allows data differences to be augmented with the utmost clarity in a completely balanced fashion. Auto-scaling is a mandated first data transformation in general PCA. For further details see, for example, chapter 5 in Reference 5.

Sampling case study: multivariate data analysis and visualisation

Figure 6 is a multivariate PCA *projection* rendition of the data in Table 1 and 2 (*score plot*), showing the mutual relationships of all field samples at a glance. The commercial samples contrast markedly with the six published ash compositions. These two main groups are delineated; the commercial samples are labelled and distinguished within the field experiment group (right).

Based on this visualisation it can readily be observed that these earliest published analytical results are markedly different from the field and commercial results, as witnessed by the left vs right sample clusters respectively. This represents the geologically known evolution of the general lava composition over the entire eruption duration.

If one wishes to study further why this is so, in the "language" expressed by the variable correlations, the accompanying *loading plot* (Figure 6B) outlines those variables for which these two sample groups display relatively high vs low concentration levels. Thus, the published results are relatively high in the concentration of elements FeO_2, MnO, P_2O_5, Ba, Cu, Na_2O, Zn and TiO_2, while the field and commercial samples display relatively low concentration levels in these elements. Some, but not all, of these relationships can be easily reconciled with standard geochemical basaltic magma differentiation understandings, but this issue need not be pursued further here.

For the present purpose we proceed to study in more detail the relationship between the field experiment samples and the closely related commercial samples only. These focused relationships are displayed in Figure 7A and B.

In Figure 7A (*score plot*) all field samples and their commercially counterparts can be readily compared in the most discriminating fashion in comparison with the absolute analytical results shown in Table 2. It is notable that field sampling group 3 sampled material which is maximally different from the commercial compositions (this difference can be put on a quantitative basis by reference to Table 1), with the preponderance of results from most of the other field sampling groups *lining up* along principal component No. 1, PC_1. Ordering this polarity feature with respect to decreasing similarity with group 3, ranks sampling groups Nos. 3, 1, 8, 2, 6, 11, 9.

The most notable among the ten field sampler groups would appear to be groups 7 and 10, which set themselves off in a different fashion, such that this feature is modelled by the second principal component, PC_2. When interpreting complementary score, and loading plot relationships, the relative proportions of the total data variance (in Table 2) must always be kept in mind, in the present case the PC_1 variability account for more than four times the variance modelled along PC_2 (69 % vs 15 %, respectively). Thus, the deviations reflecting groups 7 and 10 must be seen, and assessed, in this moderating light.

Figure 7A also delineates the variability due to the two laboratory triplicate aliquots replications, intended to reveal the sum-total of the specific aliquot extraction-plus-analysis error effects. Figure 7A reveals that the variability of these laboratory uncertainties (square boxes) are only responsible for a minor part of the total between-sample variability along both PC_1 and PC_2 axes, which collectively accounts for 84 % of the total data variance. Variations larger than these box dimensions must therefore be ascribed to differences between the ten sampler groups, given the information from Tables 1 and 2 that the local ash fall is of relatively stable composition.

It is highly relevant also to take a look at the variable relationships corresponding to Figure 7A (*score plot*), which are given in Figure 7B (*loading plot*). This data set gives rise to a practically 100 % understandable variable correlation signature for all 17 variables, fully consistent with conventional volcanic and geochemical knowledge: The "silicic" composition (SiO_2, K_2O, Na_2O, Ba) vs the anti-correlated "mafic" composition (MgO, CaO, FeO_2, Ni, Cr) polarity is a well-known geochemical feature of basaltic volcanic lavas, which lends credibility to the relevance and quality of the sampling undertaken.

Discussion and conclusions

As to the *confounding* factor described above, it is not possible to distinguish with complete resolution between the effects from material heterogeneity and the somewhat different composite sampling procedures used (from 3 to 10 increments for the specific composite samples). Indeed, there is also the possibility that one or more of the sampler groups accidentally tapped into the *sub-drill* material, which will have affected the composite sample compositions to a varying, but significant degree. Indeed, this *could* have happened for each individual increment used. Could such, for example, be the reason behind the most deviating sampler groups 7 and 10?

The value of incorporating relevant elements of the discipline of Design of Experiments (DoE) was illustrated in the results interpretation above, e.g. the value of a small embedded experimental design of triplicate replications of the final aliquot sampling step. DoE is most often applied in situations in which the experimental factors can be fully controlled, but it can also be creatively carried over to many other scenarios. Thus, the present simple aliquot replications showed up clearly in the multivariate projection plots, allowing to take in a measure of discrimination between the specific analytical uncertainty effects vs the complete between-sampler group variability.

Figure 8. A sampling group with "2 × 60 years", in field and birthday party mode.

The reason this article has traced what may appear as very detailed geological issues in such detail is that these are actually, or mimic well, very general features that attend *all* primary sampling of significantly heterogeneous materials in practice. Many lots display a similar degree of complex origin (temporal, spatial), also, lots defined as targets for environmental studies, natural processes, biological materials within agriculture, wine making etc.[10]

By multivariate PCA projection it was possible to obtain a complete overview of all essential similarities and differences between samples and in the present specific case, also between all field sampling groups.[b] These groups are of course anonymous, and shall remain so, only identified by numbers. It matters not what is the identity of the sampler groups shown in Figure 2 and the corresponding results shown in Figure 7. What matters is the degree of sampling variability shown by a group of interested, willing and eager samplers, but with significantly varying TOS competence and experience difference after all other factors have been optimally controlled. The realism of the experiment is poignant and highly relevant. Field sampling of lots of complex origin and significant heterogeneity is no walk in the park; TOS is critically needed for all *sampling processes* that aspired to representativity—regardless of the specific nature of the target lot.

What also matters is the *possibility* of augmented interpretations by taking into account all variables simultaneously—no less than 84 % of the total variability for 17 variables is captured by the graphical

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[b] In this context, it is perhaps of relevance that field sampling groups 2, 3 and 9 are the groups supposed to know their geology and sampling business well.

rendition based on only the two first principal components. What is evident from this data analysis is that there is *prima facie* close general similarity between all the field samples and the commercially obtained samples, Figure 6, but also that it is still possible to trace out and interpret highly detailed relationships *between* alternative sampling groups, a few of which would appear to perhaps have gone somewhat astray (or more likely, simply "sampled a bit too deep") based on what in "absolute" concentration units (percentage points) constitute only very small differences, cf. Tables 1 and 2. Such is the power of relevant multivariate data analysis (*chemometrics*), which in some aspects rather is a hidden elephant in the room. While many sampling tasks can be optimised by identifying the singular variable (compound, analyte) for which the lot heterogeneity is the largest, there also exist many other cases in which this is not known in beforehand, and where a simultaneous overview of the full variable correlation make-up can yield interesting pathways to new understandings and scientific/technological problem formulation.

Lessons learned

1) The Eyjafjellajökull field experiment illustrates sampling of a non-industrial heterogeneous lot with a complex origin, viz. the geological formation of the local 2010 volcanic eruption ash fall. This lot displays many potentially confounding characteristics and features. The specific field activity shows how a dedicated experiment can be designed for those sampling factors and sampling conditions that can controlled individually—laying bare only the ultimate, unavoidable confounding between degree of lot heterogeneity × sampling method.
2) There is power in adding the discipline of DoE to the professional sampler's arsenal, allowing to *design* the entire sampling campaign and/or place *embedded* experiments within any sampling superstructure, see, for example, chapter 11 in Reference 5.
3) The versatility of the RE approach could be illustrated with particular clarity. Regardless of how many sampling factors, conditions and stages involved before analysis, a RE "from the top", i.e. replicating the primary sampling will *per force* pick up effects from all active sampling errors (from all sampling stages), which is exactly what is the objective. The RE is described, for example, in chapter 9 in Reference 5 and in Reference 8.
4) Multivariate data analysis (*chemometrics*) is a further empowerment for samplers, allowing the overview, at a glance, of the relative sampling variabilities, e.g., from aliquot-extraction-plus-analysis vs the total sampling variance, Figures 6 and 7. A multivariate data analysis overview will also allow samplers to isolate the candidate analyte with the largest heterogeneity manifestation—and will depict which other analytes are correlated/anti-correlated/not correlated with, allowing a first foray into a multivariate work modus for TOS.
5) Multivariate data analysis allows extracting inherent correlation features between samples and analytes with the outmost clarity and precision, even in the case of great similarity between analytical data due to the convenient data analytical "auto-scaling" facility.

Acknowledgements

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Finally this publication is dedicated to life-long friendships, many of which are featured above; a very special recognition to soul-mate, equally "young", Guttorm Isaksson of Tromsø fame.

References

1. M.T. Gudmundsson, R. Pedersen, K. Vogfjörd, B. Thorbjarnadóttir, S. Jakobsdóttir and M.J. Roberts, "Eruptions of Eyjafjallajökull Volcano, Iceland", *EOS* **91(21),** (2010). https://doi.org/10.1029/2010EO210002
2. F. Sigmundsson, S. Hreinsdóttir, A. Hooper, Th. Árnadóttir, R. Pedersen, M.J. Roberts, N. Óskarsson, A. Auriac, J. Decriem, P. Einarsson, H. Geirsson, M. Hensch, B.G. Ófeigsson, E. Sturkell, H. Sveinbjörnsson and K.L. Feigl, "Intrusion triggering of the 2010 Eyjafjallajökull explosive eruption", *Nature* **468,** 426–430 (2010). doi: https://doi.org/10.1038/nature09558
3. R. Pedersen, *Eyjafjellajökjull: Vulkanen der Lammede Europa*. Gyldendal (2014). ISBN: 978-87-02-10062-4
4. K.H. Esbensen, "Pierre Gy (1924–2015): the key concept of sampling errors", *Spectrosc. Europe* **30(4),** 25–28 (2018). https://www.spectroscopyeurope.com/sampling/pierre-gy-1924%E2%80%932015-key-concept-sampling-errors
5. K.H. Esbensen and B. Swarbrick, *Multivariate Date Analysis—An introduction to Multivariate Data Analysis, Process Analytical Technology and Quality by Design*, 6th Edn. CAMO Software AS (2018). ISBN 978-82-691104-0-1
6. *DS 3077. Representative Sampling—Horizontal Standard*. Danish Standards (2013). www.ds.dk
7. *Get Your Sampling Right: An entry-level introduction to representative sampling*. A collection of the Sampling Columns published in *Spectroscopy Europe* (2018). https://www.spectroscopyeurope.com/get-your-sampling-right
8. K.H. Esbensen and C. Wagner, "Sampling quality assessment: the replication experiment", *Spectrosc. Europe* **28(1),** 20–25 (2016). https://www.spectroscopyeurope.com/sampling/sampling-quality-assessment-replication-experiment
9. K.H. Esbensen and C. Wagner, "Theory of Sampling (TOS) vs Measurement Uncertainty (MU) – a call for integration", *Trends Anal. Chem.* **57,** 93–106 (2014). https://doi.org/10.1016/j.trac.2014.02.007
10. K.H. Esbensen, "Theory of Sampling – what's next?", in *Proceedings of the 8th International Conference on Sampling and Blending*. AusIMM, pp. 29–38. (2017). ISBN: 978-1-925100-56-3

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The costs of sampling errors and bias to the mining industry

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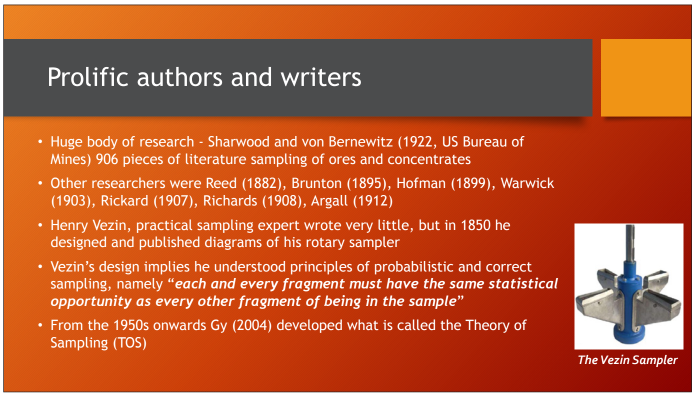
This is Richard Minnitt's regular contributing presentation at WCSB8, Perth. This presentation is another example of a subject matter that lends itself eminently to graphic rendition, which is the exact reason it was decided also to bring this feature in its original PowerPoint format; the presentation layout and style is overwhelmingly pleasing. There is here a wealth of information regarding an issue which is often lamented as lacking: what are the economic costs of inferior sampling. Richard Minnitt here collects a range of illustrative examples that will serve well for all samplers trying to convey the everlasting massage: "It pays to pay attention to unnecessary sampling errors and—bias".

The costs of sampling errors and bias to the mining industry

R.C.A. Minnitt, School of Mining Engineering, University of the Witwatersrand, Private Bag 3, 2050. Mobile: 082 481 2357; email: Richard.Minnitt@wits.ac.za

Its all about the money

- Research into the theory and practice of particulate sampling, 1850 to 1930, motivated by incorrect payments for traded commodities in USA and UK
- Substantial financial implications and scale of potential losses through poor sampling
- Sampling errors and bias highlighted the need for accurate assays of ores, concentrates, and coals
- Researchers knew of errors and bias but did not specify source



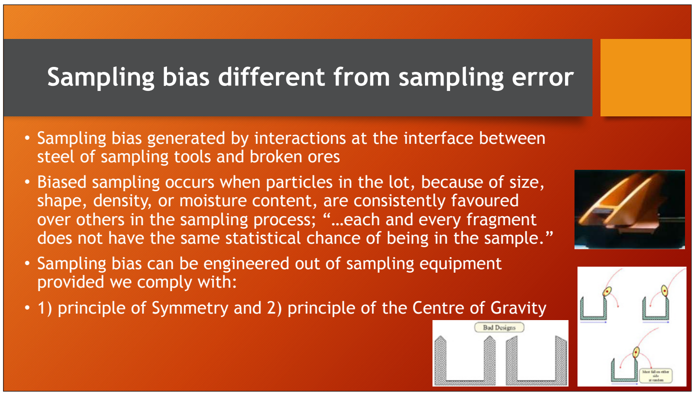
Prolific authors and writers

- Huge body of research - Sharwood and von Bernewitz (1922, US Bureau of Mines) 906 pieces of literature sampling of ores and concentrates
- Other researchers were Reed (1882), Brunton (1895), Hofman (1899), Warwick (1903), Rickard (1907), Richards (1908), Argall (1912)
- Henry Vezin, practical sampling expert wrote very little, but in 1850 he designed and published diagrams of his rotary sampler
- Vezin's design implies he understood principles of probabilistic and correct sampling, namely ***"each and every fragment must have the same statistical opportunity as every other fragment of being in the sample"***
- From the 1950s onwards Gy (2004) developed what is called the Theory of Sampling (TOS)

The Vezin Sampler

Source and nature of sampling errors

Sources of error	Name of error	Nature of error
Material characterization	*In-situ Nugget Effect (INE)*	*True error*
	Fundamental Sampling Error (FSE)	*True error*
	Grouping and Segregation Error (GSE)	*Bias*
Sampling equipment and materials handling	*Delineation Error (DE)*	*Bias*
	Extraction Error (EE)	*Bias*
	Preparation Error (PE)	*Bias*
	Weighing Error (WE)	*Bias*
Plant process and procedures	*Continuous Selection Error (CSE)*	*Error and Bias*
Analytical processes	*Analytical Error (AE)*	*Error and bias*

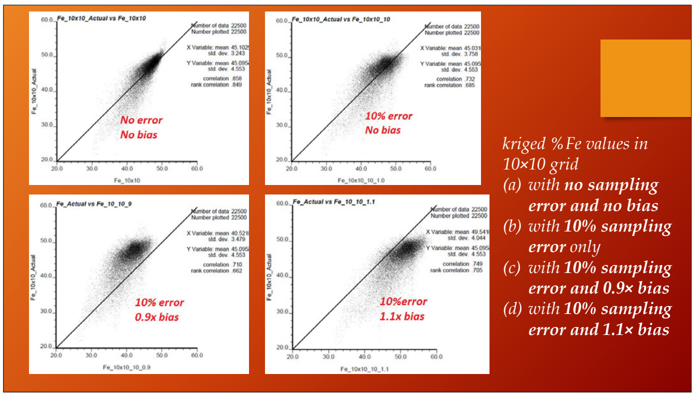


Sampling bias different from sampling error

- Sampling bias generated by interactions at the interface between steel of sampling tools and broken ores
- Biased sampling occurs when particles in the lot, because of size, shape, density, or moisture content, are consistently favoured over others in the sampling process; "…each and every fragment does not have the same statistical chance of being in the sample."
- Sampling bias can be engineered out of sampling equipment provided we comply with:
- 1) principle of Symmetry and 2) principle of the Centre of Gravity

Following that brief introduction to sampling

- How does ***sampling bias*** on grade of iron ore ***affect the revenues obtained*** for this product



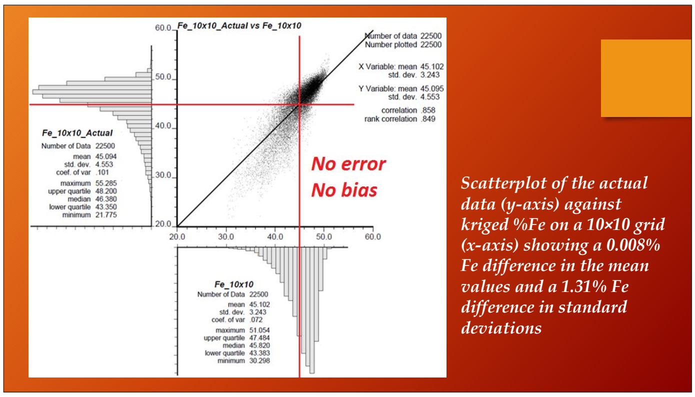
kriged %Fe values in 10×10 grid

(a) with no sampling error and no bias

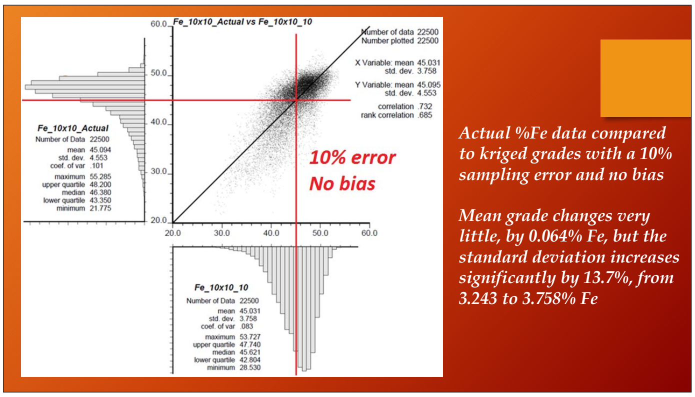
(b) with 10% sampling error only

(c) with 10% sampling error and 0.9× bias

(d) with 10% sampling error and 1.1× bias

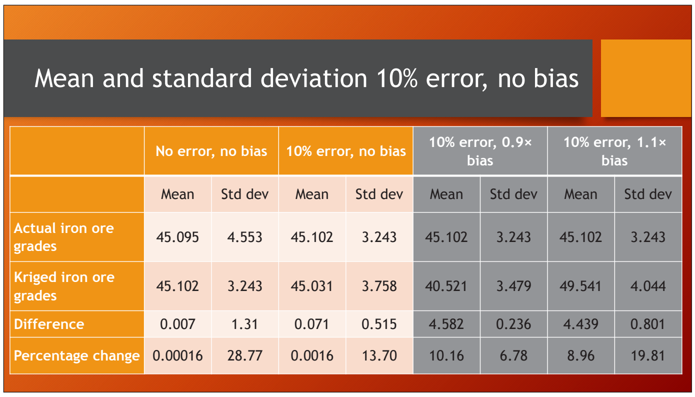


Scatterplot of the actual data (y-axis) against kriged %Fe on a 10×10 grid (x-axis) showing a 0.008% Fe difference in the mean values and a 1.31% Fe difference in standard deviations



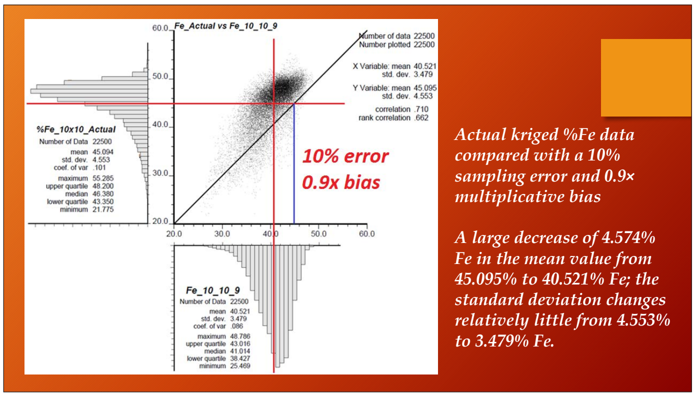
Actual %Fe data compared to kriged grades with a 10% sampling error and no bias

Mean grade changes very little, by 0.064% Fe, but the standard deviation increases significantly by 13.7%, from 3.243 to 3.758% Fe



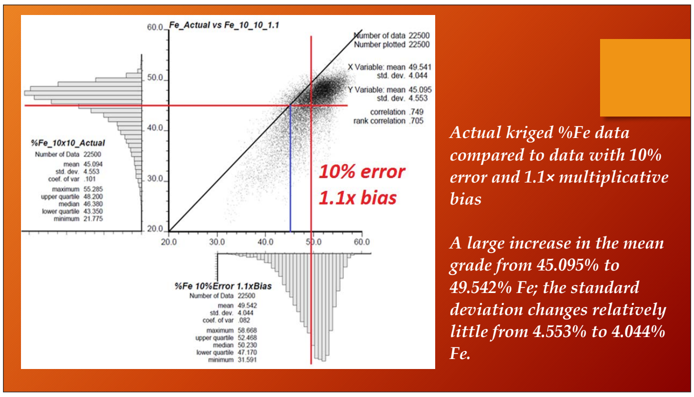
Mean and standard deviation 10% error, no bias

	No error, no bias		10% error, no bias		10% error, 0.9× bias		10% error, 1.1× bias	
	Mean	Std dev	Mean	Std dev	Mean	Std dev	Mean	Std dev
Actual iron ore grades	45.095	4.553	45.102	3.243	45.102	3.243	45.102	3.243
Kriged iron ore grades	45.102	3.243	45.031	3.758	40.521	3.479	49.541	4.044
Difference	0.007	1.31	0.071	0.515	4.582	0.236	4.439	0.801
Percentage change	0.00016	28.77	0.0016	13.70	10.16	6.78	8.96	19.81



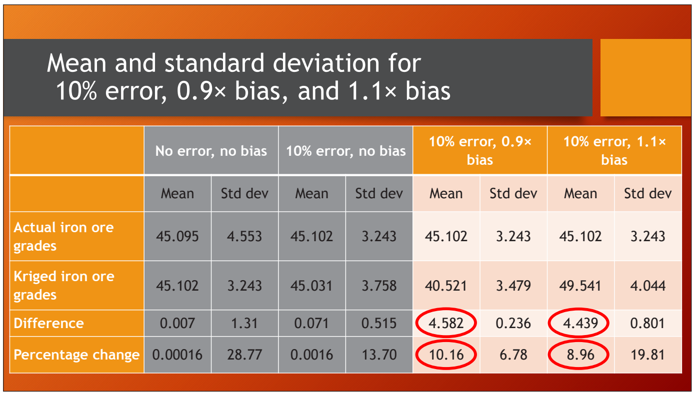
Actual kriged %Fe data compared with a 10% sampling error and 0.9× multiplicative bias

A large decrease of 4.574% Fe in the mean value from 45.095% to 40.521% Fe; the standard deviation changes relatively little from 4.553% to 3.479% Fe.



Actual kriged %Fe data compared to data with 10% error and 1.1× multiplicative bias

A large increase in the mean grade from 45.095% to 49.542% Fe; the standard deviation changes relatively little from 4.553% to 4.044% Fe.



Mean and standard deviation for 10% error, 0.9× bias, and 1.1× bias

	No error, no bias		10% error, no bias		10% error, 0.9× bias		10% error, 1.1× bias	
	Mean	Std dev	Mean	Std dev	Mean	Std dev	Mean	Std dev
Actual iron ore grades	45.095	4.553	45.102	3.243	45.102	3.243	45.102	3.243
Kriged iron ore grades	45.102	3.243	45.031	3.758	40.521	3.479	49.541	4.044
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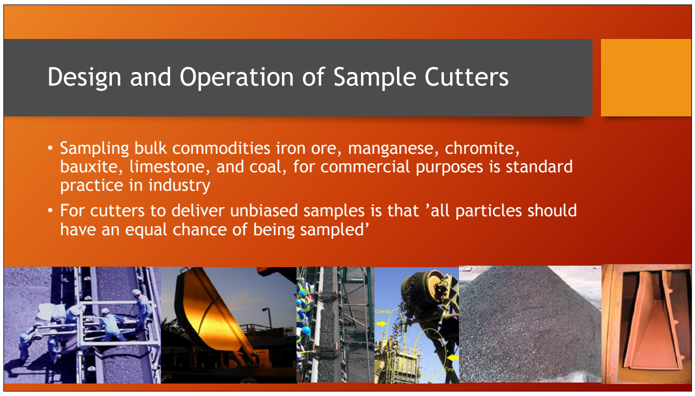
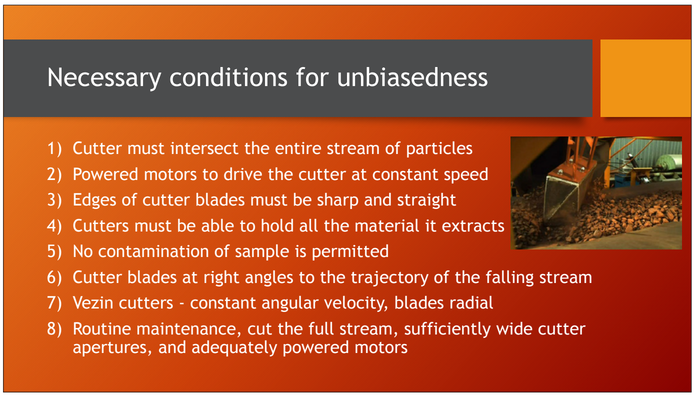


Design and Operation of Sample Cutters

- Sampling bulk commodities iron ore, manganese, chromite, bauxite, limestone, and coal, for commercial purposes is standard practice in industry
- For cutters to deliver unbiased samples is that 'all particles should have an equal chance of being sampled'



Necessary conditions for unbiasedness

1) Cutter must intersect the entire stream of particles
2) Powered motors to drive the cutter at constant speed
3) Edges of cutter blades must be sharp and straight
4) Cutters must be able to hold all the material it extracts
5) No contamination of sample is permitted
6) Cutter blades at right angles to the trajectory of the falling stream
7) Vezin cutters - constant angular velocity, blades radial
8) Routine maintenance, cut the full stream, sufficiently wide cutter apertures, and adequately powered motors

How will *bias affect the price* they receive?

- Saldanha Bay iron ore facility
- Loading 100000 tonnes of iron ore
- Taking a 21 tonne composite sample

Particle size distribution, particle volume, particle mass, mass of size fraction in increment, mass of size fraction, and possible number of particles in a 21 tonne composite sample with an average grade of 63.805% Fe

Size fractions	Diameter of largest particle	Radius	Volume of a particle (if a cube)	Mass of one particle in g	Percentage mass distribution of a typical lump ore	Mass of size fraction in sample	Mass of size fraction in gram	Possible particles in sample	Fe of size fraction	Fe of total sample
+31.5 mm	31,5	15,75	0,00001636	84	1	210	210 000	2 493	66,0	63,805
-31 +26.5 mm	26,5	13,25	0,00000974	50	7	1470	1 470 000	29 309	65,5	
-26.5 +25.0 mm	25	12,5	0,00000818	42	5	1050	1 050 000	24 934	65,0	
-25 +20.0 mm	20	10	0,00000419	22	21	4410	4 410 000	204 533	64,5	
-20 +16.0 mm	16	8	0,00000214	11	22	4620	4 620 000	418 501	64,0	
-16 +12.5 mm	12,5	6,25	0,00000102	5	18	3780	3 780 000	718 085	63,5	
-12.5 +10.0 mm	10	5	0,00000052	3	12	2520	2 520 000	935 007	63,0	
-10 +8.0 mm	8	4	0,00000027	1	7	1470	1 470 000	1 065 275	62,5	
-8 +6.3 mm	6,3	3,15	0,00000013	1	3	630	630 000	934 831	62,0	
-6.3 mm	5	2,5	0,00000007	0	4	840	840 000	2 493 352	61,5	
					100	21000	21 000 000			

Financial impact of sampling error and sampling bias

- +25 mm fraction tends to be missed during the sampling procedure
- Due to bias in the sampling equipment assume 25% of fragments lost from the four largest fragment sizes
- 9%of the larger fragments are under-represented in the 21 t sample reducing grade by 0.10% Fe
- Large particles lost to the sample are never actually seen because they simply fall back onto the incoming stream and continue to the loading bay of the vessel

Number and mass of +25 mm particles lost during sample extraction as a result of sampling bias and the average grade of 63.705% Fe after losing the particles

Size fractions	No of +25 mm particles lost during sampling	Mass (g) +25 mm particles lost during sampling	Remaining particles	Mass left over (kg)	Percentage left over	Fe after losing particles
+31.5 mm	623	52500	1870	157,500	0,8254	63,705
-31 +26.5 mm	7327	367500	21981	1102,5	5,7775	
-26.5 +25.0 mm	6233	262500	18700	787,5	4,1268	
-25 +20.0 mm	57269	1234800	147264	3175,2	16,6392	
-20 +16.0 mm			418501	4620	24,2104	
-16 +12.5 mm			718085	3780	19,8085	
-12.5 +10.0 mm			935007	2520	13,2057	
-10 +8.0 mm			1065275	1470	7,7033	
-8 +6.3 mm			934831	630	3,3014	
-6.3 mm			2493352	840	4,4019	
Total	71453	1917300	6 754 867	19082,7	100	

Conclusions

- Bias excludes 9% of higher grade fragments giving a grade difference of 0.10% Fe (63.805 - 63.705% Fe = 0.10%
- Bias remains the same irrespective of the mass of the composite sample
- The 0.10% Fe bias in the grade for a 100 000 t load at a price of $70 per ton and the lot grade of 63.805% Fe would amount to a loss of $10 971, not much on a load worth $7.0 million
- South Africa exports 60 Mt of iron ore on 600 ships annually
- Cumulative losses per annum could be as much as $6,6 million

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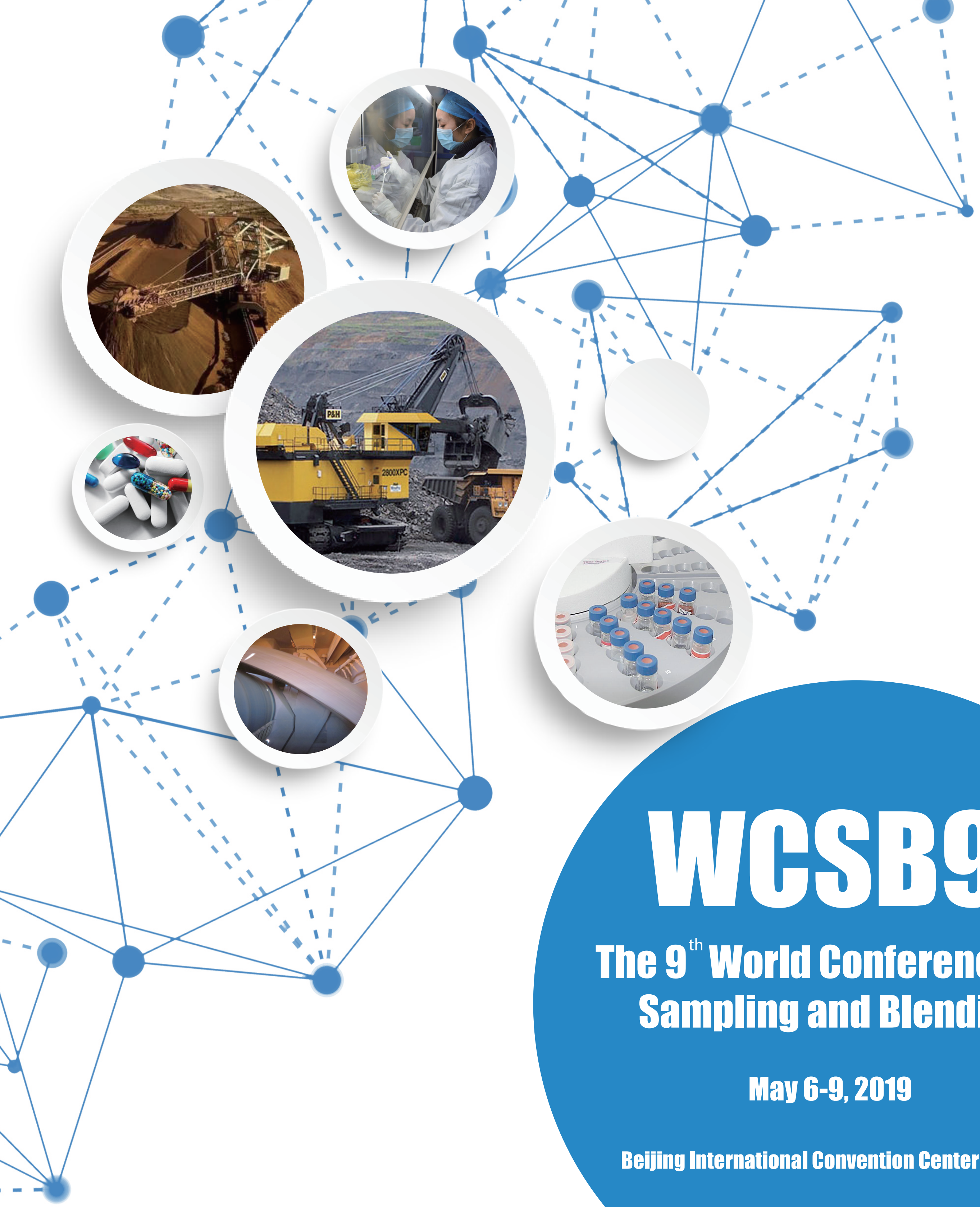
- All are gratefully acknowledged.



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Hosted by the BGRIMM Technology Group (former Beijing General Research Institute of Mining & Metallurgy), the 9th World Conference of Sampling and Blending (WCSB9) will be held in Beijing on May 6-9, 2019.

The conference aims to cover current research and theory in the field of sampling and blending, and share the latest technological knowledge, operational practices and advances in the field. The application areas covered by the conference will include mining, cement, food, feed, pharmaceuticals, agriculture, biology, and environmental protection. The last 8 conferences have been held in Denmark, Australia, Brazil, South Africa, Chile, Peru and France, and now WCSB9 comes to China in 2019.

Why attend

- The conference is the top level international sampling and blending event, sharing the latest knowledge and industry practices;
- Papers will cover a wide range of topics, including minerals, metals & alloys, cement, pharmaceuticals, food, agricultural & biomass products, environmental monitoring, etc;
- Gain valuable knowledge on sampling and blending from respected international experts in the field;
- Expand your sampling and blending contacts and strengthen cooperation on a worldwide basis;
- Improve your personal knowledge through attendance at associated conference workshops;
- Visit the most advanced laboratories in China.

Who should attend

Representatives from Universities and research institutes, mining and trading companies, instrument manufacturers and testing laboratories, regulatory authorities, international quality associations, and government bodies, as well as grade and quality control staff, risk assessors and risk managers.

Theme of the Conference

Smart Sampling & Blending

Main Topics

- Case studies
- Development of national and international standards
- Theory of sampling and blending
- Sampling and blending in various areas, including minerals, metals and alloys, pharmaceuticals, food, feed, agriculture, biomass products, and environmental monitoring
- Quality assurance and quality control (QAQC)
- Metallurgical accounting
- New developments in sampling, sample preparation and blending equipment
- Analytical process technology
- Future technologies

Exhibition Scope

- Sampling, sample preparation and blending equipment
- Automatic online monitoring equipment
- Third party inspection and certification laboratory
- Laboratory analytical instrument and technology
- Testing, measurement and quality controlling
- Mining machinery and equipment
- Environmental sampling and monitoring
- Information consultation & technical service
- Others

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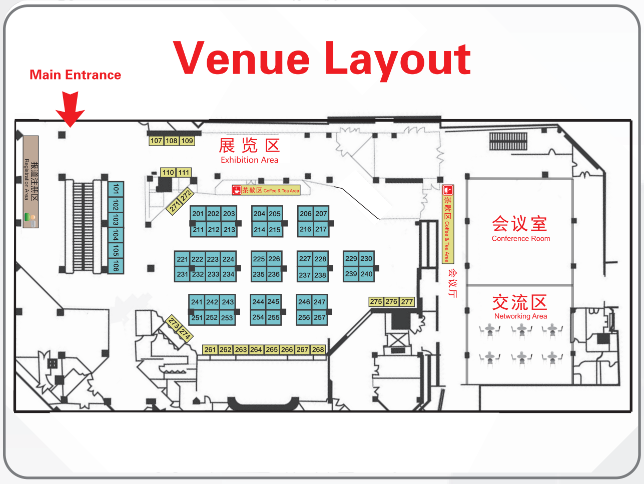
A trade exhibition will be held at the conference venue, which will provide companies with an excellent opportunity to display their products and services to delegates. A number of pre- and post-conference workshops will also be organized on relevant topics, including sampling theory and practice.

Schedule for Submission of Papers

- Closing date for receipt of abstracts November 9, 2018
- Notification of acceptance of abstracts November 26, 2018
- Receipt of first draft of papers December 21, 2018
- Referred papers returned to authors February 28, 2019
- All final papers received March 21, 2019
- Publication of Proceedings April 19, 2019
- WCSB9 May 7, 2019



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Pass Types	Registration Types	Price（RMB）		Amount
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All Access Pass	General Delegate	RMB 5,500	RMB 6,000	
	Author of the Conference Paper	RMB 5,500		
	Relevant Association Member	RMB 5,500		
	Student	RMB 2,000		
The package for all access pass include: delegate badge, delegate bag, notebook, pen, electronic conference proceedings , conference guide, lunch ticket, tea & coffee break, cocktail party and Gala dinner.				
One Day Pass	General Delegate	RMB 3,000		
	Relevant Association Member	RMB 2,500		
The package for one day pass include: delegate badge (one day only), delegate bag, notebook, pen, electronic conference proceedings , conference guide, tea & coffee break and lunch ticket.				
Post-Event Tour	Route	Select	Price(RMB)	Amount
	Route 1, R&D Center of BGRIMM Technology Group + The Great Wall & Route 2, The National Palace Museum + R&D Center of BGRIMM Technology Group	☐Route 1 ☐Route 2	RMB 450/person	

Participant List:

Delegate	Position	Tel	Email	Department

Sponsorship and Advertising Opportunities:

The 9th World Conference of Sampling and Blending provides unique business opportunities for company and institute to brand marketing and communication with target clients. Improve your exposure and visibility before, during and after the event by taking advantage of event sponsorship package and advertising opportunities.

Major Sponsorship

☐ Platinum Sponsor RMB 150,000 ☐ Gold Sponsor RMB 120,000 ☐ Silver Sponsor RMB 80,000

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☐ Gala Dinner Sponsor RMB 200,000
☐ Delegate Bag Sponsor RMB 100,000
☐ Coffee & Tea Sponsor (3 days) RMB 50,000
☐ Delegate Badge Sponsor RMB 60,000
☐ Gala Dinner Drink Sponsor RMB 50,000
☐ Lunch Sponsor (3 times) RMB 80,000
☐ Delegate Notebook Sponsor RMB 60,000
☐ Delegate Lanyard Sponsor RMB 60,000
☐ Workshop Sponsor RMB 100,000
☐ Water Sponsor RMB 80,000
☐ Delegate Pen Sponsor RMB 60,000
☐ Session Sponsor RMB 60,000

Advertising Opportunities

☐ Event Catalogue Inner Pages	210mm X 285mm	RMB 8,000
☐ Event Catalogue Inside Cover	210mm X 285mm	RMB 15,000
☐ Event Catalogue Inside Back Cover	210mm X 285mm	RMB 15,000
☐ Bag Insert (A4 front and back page)		RMB 15,000

1.Please duplicate this form if you have more participants, please be sure to fill in the correct email address for the latest conference information
2.Cancellation will only be refunded, less a 40% administration fee, if a written request is received before February 7th, 2019, Cancellation received after February 2nd ,2019 will not be refunded.

The Organizing Committee of WCSB9

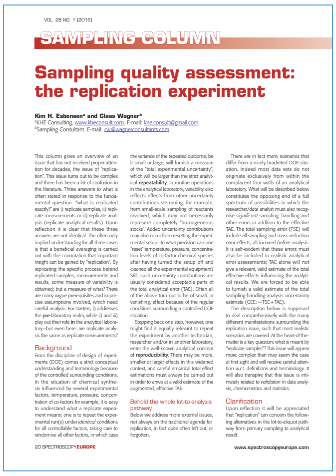
Tel: +86 10 6446 5066 Fax: +86 10 6446 6038 Email: info@chinamining-expo.org Web: www.wcsb9.com

Sampling Resources

Sampling Columns in *Spectroscopy Europe*

SPECTROSCOPY europe

The essential magazine for spectroscopists in Europe



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SAMPLING COLUMN

Sampling quality assessment: the replication experiment

Kim H. Esbensen and Claas Wagner

The Sampling Columns published in the free magazine, *Spectroscopy Europe*, and edited by Kim Esbensen and Claas Wagner are a valuable introduction to representative sampling and the Theory of Sampling (TOS).

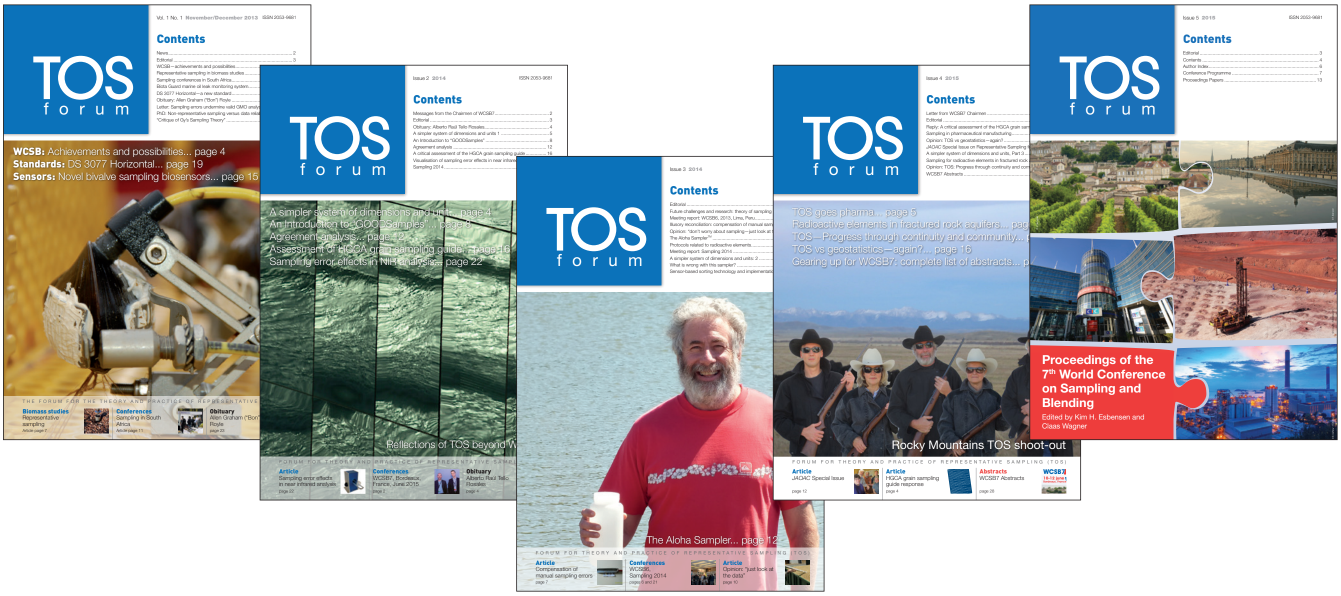
All can be read free-of-charge in print, web and digital editions, as well apps for iOS and Android devices.

Starting with an introduction to TOS, the columns have continued by looking at heterogeneity, composite sampling, a sampling quality assessment and sampling quality criteria.

Read all the Sampling Columns at:

http://www.spectroscopyeurope.com/sampling

TOS forum



TOS forum

WCSB: Achievements and possibilities... page 4
Standards: DS 3077 Horizontal... page 19
Sensors: Novel bivalve sampling biosensors... page 15

Reflections of TOS beyond W

The Aloha Sampler... page 12

Rocky Mountains TOS shoot-out

Proceedings of the 7th World Conference on Sampling and Blending
Edited by Kim H. Esbensen and Claas Wagner

Read all the back issues of *TOS forum* at:

https://www.impopen.com/tosf

Recent chemometric applications in the geoscience, technology and industry:

K.H. Esbensen, N.H. Schovsbo and L. Kristensen, Down-Hole Permeability Prediction—A Chemometric Wire-Line Log Feasibility Study from a North Sea Chalk Well.

N.H. Schovsbo, H. Holmslykke, C. Kjøller, L. Kristensen, K.H. Esbensen, A.Y. Halim, S.N. Gottfredsen, K.L. Feiberg and S.M. Nielsen, "Decoding the water flooding process from produced water composition—a case study from the Halfdan chalk oil field", *Danish North Sea 79th EAGE Conference and Exhibition* (2017). doi: 10.3997/2214-4609.201701295

R. Romanach and K.H. Esbensen, "Theory of Sampling (TOS) for development of spectroscopic calibration models", *Amer. Pharmaceut. Rev.* Spectroscopy/ Review section, pp. 1–3 (September/November 2016).

N.H. Schovsbo, H.D. Holmslykke, C. Kjøller, K. Hedegård, L. Kristensen, E. Thomsen and K.H. Esbensen, "Types of formation water and produced water types in Danish oil and gas fields: Implications for enhanced oil recovery by 'smart water' injection", *Geol. Surv. Denmark Greenland Bull.* No. 35, pp. 43–46 (March 2016). http://www.eng.geus.dk/media/10857/nr35_p43-46.pdf