

# TOS

f o r u m

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FORUM FOR THEORY AND PRACTICE OF REPRESENTATIVE SAMPLING (TOS)

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## Messages from the Chairmen of WCSB7

Philippe Davin and Stephane Brochot

At the conclusion of WCSB6 an executive meeting elected two new co-chairmen for WCSB7, who have decided to keep us all updated with relevant information in this column. Co-chairmen Philippe Davin and Stephane Brochot are pleased to announce that the 7<sup>th</sup> World Conference on Sampling and Blending (WCSB7) will take place in Bordeaux, France, in the week of 8–12 June 2015. The conference itself will take place during the three latter days, 10–12 June, while short courses will be offered for the beginning of the week, 8 and 9 June. The conference venue will be: Centre de Congrès Cité Mondiale, 18 parvis des Chartrons, 33 080 Bordeaux, France. Latitude: N 44° 50' 59.87" / Longitude: W 0° 34' 16.09".

### Bordeaux

Bordeaux is of course a city very well known for its wines, but did you know that it is also a city of art and history, indeed listed as a UNESCO World Heritage Site! Discover more about Bordeaux: <http://www.bordeaux-tourisme.com/pl/index.pl?langueSelected=uk>

The city offers a large selection of hotels at all price levels. The chairmen will supply

more information of the selected conference hotels (and their deals) in the next issue of *TOS forum*.

### Association WCSB7

An *ad hoc* association, "Association WCSB7", has been created under French law in order to organise this event in the most efficient way. Association WCSB7 will be the *de facto* organiser of the conference. In order to be able to carry out all organisational, administrative and practical work leading up to the conference week, funding is needed. Funding in the next 14 months can be considered as advanced payment of conference fees and/or sponsorship. Should you be interested in helping the association already now (**greatly appreciated**), please contact [wcsb7@caspeo.net](mailto:wcsb7@caspeo.net).

We also invite you to join the association personally by contacting [wcsb7@caspeo.net](mailto:wcsb7@caspeo.net)

### Proceedings—publication

Two experienced gentlemen in scientific publishing, Professor Kim H. Esbensen (editor for the WCSB1 Proceedings) and Professor Emeritus Pentti O. Minkkinen (past Editor for *Chemometrics and*



*Intelligent Laboratory Systems* and co-editor of the WCSB1 Proceedings) have offered to organise publication of the WCSB7 proceedings, bringing them in line with current trends regarding on-line publishing (with concomitant significant savings on the conference budget). They report that several options are currently under negotiations with a reputable publishing house (print/on-line), but it should calm everybody's concerns to know that it is a requirement that conference delegates shall receive a copy of all proceedings *at the conference*, and that a peer-reviewed selection of papers will also be published in *Chemometrics and Intelligent Laboratory Systems* in order to make the conference and its achievements known and available to a larger audience within science, technology and industry. It is reported that negotiations are progressing well; *Messieurs* Esbensen & Minkkinen will report in full in the next issue of *TOS forum*.



City of Bordeaux. UNESCO World Heritage Site.

## Welcome to the second issue of *TOS forum*

It is a very satisfactory achievement that you are now in possession of the second issue of *TOS forum*. The objective is to develop the forum into a regular periodical, and we are well under way. I estimate that we shall have published *TOS forum* Issue 5 before the next World Conference on Sampling and Blending, which takes place in Bordeaux, June 2015—see “Messages from the Chairmen WCSB7” opposite.

The interest in publishing minor progress reports from both academic and industry projects in *TOS forum* is growing in a satisfactory manner (the table of contents is already half full for *TOS forum* Issue 3), while several other types of presentations relate to the express need for effective communication between all members of the world community of professional samplers. *TOS forum* was created precisely with these needs and desires in mind; so far developments could not be better.

I have a serious issue to discuss with all our readers, an issue that must not cause misunderstanding. The editor has received

a couple of *complaints*: “There is hardly anything of interest for the mining world to be found in *TOS forum*”. This is both true—and this is not true (*sic*).

**True**, most likely—undoubtedly—because the entire mining community has just been well served by WCSB6, November 2014, Lima. Everyone that had anything important to communicate to our community had his/her say in Lima. It is still very early days after this successful conference—and more important: **all** types of contributions are welcome in *TOS forum*. Anyone from the mining industry who has anything *more/new/complementary* to the proceedings facility from WCSB6 to say... please just start clicking away on your keyboard. Your mining-related contribution is no farther away from *TOS forum* than an e-mail to [ke@geus.dk](mailto:ke@geus.dk)—I extend a serious and warm invitation to all.

**Not true**, because the quite satisfactory spread of theoretical and application pieces in *TOS forum* Issues 1 and 2 are of meant

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The front page photo may puzzle you at first, but it is subtly connected to *TOS*, however. You are looking at the façade of the Lima Marriott Hotel, the venue for WCSB6 (2013).  
Photo: Anne Jodon Cole

### Be part of the next issue of *TOS forum*!

We welcome contributions to *TOS forum*: articles, letters, comment, news or news of PhD projects for the PhD Presentations column.

*TOS forum* Editor, Kim Esbensen, would be pleased to discuss any ideas you may have and to receive your contributions.

#### EDITOR

Kim H. Esbensen (Geological Survey of Denmark and Greenland (GEUS), Copenhagen, and ACABS research group, Aalborg University, campus Esbjerg, Denmark; [ke@geus.dk](mailto:ke@geus.dk))

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Presentation of the 2013 Pierre GY Sampling Gold Medal at WCSB6, 24 November 2013, Lima.

*TOS forum* will be available on subscription in 2015. Visit [www.impublications.com/tos-forum](http://www.impublications.com/tos-forum) for details.

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## Alberto Raúl Tello Rosales (1951–2014)

Francis Pittard

Alberto Raúl Tello Rosales, born 3 January 1951 in Valparaíso, Chile died suddenly at the beginning of 2014.

He was a loving husband of Pilar, caring father of two daughters and two sons, and a proud grandfather.

Alberto was a shining example of higher education in Chile:

- B.Sc. in Chemistry, University of Chile, 1973
- M.Sc. Mining Engineering, University of Chile 1991
- Diploma in Applied Statistics, University of Santiago, Chile, 1997

In 1973 Alberto started to work at the Mining and Metallurgical Research Center, CIMM in Chile, where he built an impressive experience for 20 years in the mining industry and acquired in-depth knowledge of technical processes, quality systems and process control.

In 1995, Alberto started his consultancy career in the areas of Quality Systems, Analytical Chemistry, Process Control and Sampling. In association with Francis Pittard Sampling Consultants LLC, Alberto gave many sampling courses in Chile, Peru and Venezuela. He provided consultancy to national and international mining and metallurgical companies primarily working in Chile and in Peru. His advice improved a wide range of processes from mining areas to the final product in both non-metallic and precious metals recovery industries.

Alberto enjoyed playing golf, reading books and to travel around the world with his wife and family. An outdoor man and a great gardener, he specially loved being a professional chemist at work and to translate this into great cooking at home. His friends and family will always remember his gourmet dishes.



Alberto Tello with his wife Pilar

Alberto Tello will always be remembered amongst his co-workers as a great teacher, a leader as well as a team-player, a gentleman, a good friend and a highly professional colleague.

*continued from page 3*

to be of significant inspirational value for all who are interested in sampling, of course also for the mining world! It is the very same principles of representative sampling that undermines each-and-every entry in the forum. That said, the Editor shall always be hard at work seeking out new, novel, different angles and application fields where TOS shows its value—surely this type of pushing the envelope can only be of interest to absolutely all with a commitment to disseminate TOS.

While the present issue is still somewhat the result of invitations and solicitations regarding the subject-matter horizon (and while such an active role will continue on behalf of the editorial office), it is hoped that this gradually will be taken over by a drive for wanting “my contribution” in *TOS forum* as well. It is clear to the Editor and the Publisher that it is our solemn obligation to facilitate such a development, and we assure the readership that no efforts shall be spared in this regard. It is the goal that by WCSB7,

*TOS forum* has developed to be both a necessary and the sufficient imperative for a revolution in the ability for the world community of sampling to be in close contact at all times between the biannual WCSBs.

**The Editor** wishes to thank the governing body in charge of selecting recipients for the Pierre Gy Sampling Gold Medal for receiving this award at WCSB6, Lima 2014. This is an immense honour, one that cannot be surpassed in my personal scientific endeavour—ever. It is the greatest possible inspiration and drive to continue to serve the world sampling community in the best possible fashion.

Kim Esbensen

### Final words from our Publisher, IM Publications

*Putting together any publication, but especially a new one, is a lot of work, as Kim has discovered and alluded to above! Beyond*

*the content—the words and images—the important matter of commercial success needs to be considered, without which any long-term publication is impossible. There are only two sources of income: subscriptions and sponsorship. The former is familiar to us all! The latter may be direct sponsorship or advertising. TOS forum has been fortunate that sponsorship has been available to offset some of the costs in its publication and distribution so far. In the end though, TOS forum is likely to need to be of sufficient interest and value to its readers that they will subscribe. Until WCSB7, TOS forum will remain free, but at or soon after that meeting, a decision will need to be made about its longer-term viability.*

*In the meantime, we need to work to ensure that TOS forum meets the needs of the entire TOS community, but also the wider analytical community who may not appreciate fully the importance of representative sampling. I hope you will help Kim and us in our endeavours to achieve this.*

Ian Michael

# A simpler system of dimensions and units. Publication #1

Francis F. Pitard

Besides being one of the world intellectual leaders in the field of the theory and practise of representative sampling, the Theory of Sampling (TOS), Francis F. Pitard is also a prolific writer on several other subjects, a.o. having published two novels: *Heirs of a Lost Race* and its sequel: *Rapa Nui Settlers*, as well as a scientific tour-de-force arguing for a radically alternative view of the world, developed from his decade long collaboration with Charles O. Ingamells, entitled: “The Possibilities of Our Sub-Quantic Identity — The Theory of Vacuoles and a Simpler System of Dimensions and Units”, which has caused a stir in physical, history of science and philosophical circles. From this work, *TOS forum* has asked Francis to edit the last topic into a series of papers for this audience. You may perhaps wonder what such a topic has to do with sampling, with representative sampling? Please read, and be enlightened...—Editor.

Many publications show beyond any possible doubt that our current system of dimensions and units, metric or not, is not an appropriate tool for advanced Nuclear Physics and Astronomy; it is unnecessarily complex with foundations that are more emotional than scientific. The system is acceptable for our day-to-day lives, when we cook for the family or build a car, totally unacceptable when we explore what the Universe is. A much better and simpler system is suggested. Let us prove beyond any possible doubt that time, mass, permeability and permittivity do not need units of their own. In the suggested new system, all values for the “fundamental” physical constants are absolute, with the exception of the time-thickness constant. This alone eliminates unnecessary ambiguity and greatly simplifies our research for the ultimate truth about our place in this Universe.

There are flaws in standard models. All standard models have flaws. Flaws are not easy to find, otherwise they would not exist. Flaws in the current model are from a faulty dimensional system and an irrational system of units, metric or not, that do not seem appropriate for advanced Physics. A series of short publications is suggested, so the reader can slowly eliminate one of the greatest paradigms of our time that may be responsible for slow progress in Science. So, this is publication #1, and others will follow. The suggested publications are a series of adapted topics for the purpose of making progressive reviews of weaknesses in the ways we think today. Publications will start exposing simple concepts, and then getting increasingly more

complex to the point where fundamental changes in the ways we understand matter will be suggested and speculated on. This work is inspired from a textbook published in 2012 by the author and his late friend Charles O. Ingamells (ISBN: 978-0-9850631-0-8).

## Units—a history

On 10 December 1799, the French Legislative Assembly voted to define a standard of length, the “metre” as the  $E^{-7}$  part of the earth’s quadrant. For those who may not know, Napoleon Bonaparte was in charge, and you did not argue with the First Consul, a short time before he self-promoted to the title of Emperor. On the same day, the mass of a cubic decimetre of water at its maximum density was chosen as the standard of mass and was named the kilogram. The third fundamental unit, time, was determined by astronomers as  $1/86,400$  of a solar day. Thermal and electrical units are both vaguely related to this *MLT* system. The National Bureau of Standards defined the calorie as 4.184 joules. One joule is  $E^7$  cgs units of energy. The electrostatic unit of quantity of electricity is the quantity which when concentrated at a point 1 cm from an equal and similarly concentrated quantity is repelled by a force of 1 dyne. The quantity transferred by one ampere in one second is the coulomb. The *MKS/Giorgi* system of units required four fundamentals: metre, kilogram, second and ohm. The ohm is the resistance of a column of mercury of uniform cross-section having a length of 106.380 cm and a mass of 14.4521 g and a temperature of  $0^\circ\text{C}$ . A centigrade degree is  $1/100$  of the temperature rise of water on being heated

from its freezing to its boiling point under a pressure of one atmosphere, i.e. the pressure exerted by 760 mm of mercury at the surface of the earth. The ohm has also been defined as the electric resistance between two points in a conductor when a constant potential difference of one volt, when applied between these two points, produces in this conductor a current of one ampere. An ampere is  $1.0363 E^{-5}$  faraday/second or 2.9979 electrostatic cgs units. And so on and on... this is what we do science with!

The plethora of units and their definitions and the inconsistencies therein has led to innumerable congresses, bureaus, publications and passionate discussion, leading to the *SI* system today which as we will see is far from being the panacea. Long ago, Eddington proposed that the number of physical constants may eventually be reduced from five or more to only one: “I believe that the whole system of fundamental hypotheses can be replaced with epistemological principles... all the fundamental laws and constants of Physics can be deduced unambiguously from *a priori* considerations, and are therefore subjective.”

Currently accepted definitions of the various “fundamentals” appear in the International System of Units (*SI*) as follows: the metre is the length of the path travelled by light in vacuum during a time interval of  $1/299792458$  of a second. The kilogram is the mass of the International prototype of the kilogram. The second is the duration of 9,192,631,770 periods of the radiation corresponding to the transition between the two hyperfine levels of the ground state of  $^{133}\text{Cs}$  atom. The ampere is that constant current which, if maintained

in two straight parallel conductors would produce between these conductors a force equal to  $2 \times 10^{-7}$  newton per metre of length. The Kelvin, unit of thermodynamic temperature, is the fraction  $1/273.16$  of the thermodynamic temperature of the triple point of water. The mole is the amount of substance of a system which contains as many elementary entities as there are atoms in  $0.012\text{kg}$  of  $^{12}\text{C}$ . It is very hard to resist noting the “King-Henry’s Thumb” principle here, over which we did not make much improvement.

### An attempt for a simpler system of units

As mortals, we live by the clock, with life flowing through us as length  $[L]$ , mass  $[M]$  and time  $[T]$ . The commonly accepted dimensions of physical quantities are  $[L]$ ,  $[M]$  and  $[T]$ . Most ordinary things and phenomena are described in terms of these dimensions; it is our today paradigm and it is extremely difficult to think any other way; it is the way we were told to think.

If you spill mercury on a smooth floor, the liquid metal will bead into droplets of various sizes. The same happens if water is spilled on a well-waxed car roof. The small droplets are very nearly spheres. The large droplets are flattened or nearly ellipsoidal. The larger ones are flat puddles of uniform height or thickness. Using the “method of dimensions” we may calculate the depth, height or thickness of these drops from three known parameters. The height,  $h$ , depends on  $\rho$ , the density of the liquid, its surface tension,  $\gamma$  and  $g$ , the force (acceleration) due to gravity, which flattens the droplets of water or mercury. The height of the puddles is a function of these three parameters. Writing this sentence in abbreviated form gives  $h = f(\rho, \gamma, g)$ . We can state the dimensions of density, surface tension and the acceleration due to gravity on the earth’s surface in terms of length, mass and time. Density,  $\rho$ , is mass per unit volume. Volume is length times length times length, or  $[V] = [L \cdot L \cdot L] = [L^3]$ . If a brick is  $0.05\text{m}$  by  $0.08\text{m}$  by  $0.20\text{m}$ , its volume is  $0.05 \times 0.08 \times 0.20 = 0.0008\text{m}^3$ . If the brick weighs  $2\text{kg}$  on this earth (it would weigh much less on the moon), its mass is  $2\text{kg}$  and its density is  $2\text{kg}$  per  $0.0008\text{m}^3$ , or  $\rho = 2/0.0008 = 2500\text{kg m}^{-3}$ . This is one of the silly systems by which we live and it would not be a very good idea for anyone to challenge it.

Surface tension,  $\gamma$ , has dimensions  $[M/T^2]$ , and the force of gravity is due to an acceleration,  $g$ , with dimensions  $[L/T^2]$ . These statements require explanations. Acceleration is easiest to describe because everyone knows what it is! It is the rate of speed increase when you step on the car gas pedal. In a car, you may measure it in  $\text{km per hour per second}$ . In one second, you may go from  $50\text{km/h}$  to  $60\text{km/h}$ . Your acceleration,  $a$ , is  $10\text{km per hour per second}$ , with dimensions  $\text{km (length) per hour (time) per second (time)}$ : acceleration  $= [L/T^2]$ . Acceleration due to gravity,  $g$ , is the acceleration of an object that free-falls from a height onto the earth. It determines the downward force,  $F$ , that any standing object exerts on its floor.

If we put our  $0.05 \times 0.08 \times 0.20\text{m}^3$  brick on a scale, the scale registers  $2\text{kg}$ . The brick is exerting a  $2\text{-kg}$  force on the scale. If we were on the moon with the same brick and the same scale, the brick would weigh much less than  $2\text{kg}$ , but it still has the same mass!

If  $W$  is the weight of an object,  $M$  the mass of this object, and  $g$  the acceleration due to gravity of the object in free fall, we have the relationship:

$$W = M \cdot g \quad (1)$$

Therefore, if  $g$  at the surface of the earth is a reference taken as 1, then the weight and the mass appear to be the same thing; they most certainly are not.

Too often, deeply set axioms and beliefs, in exquisitely subtle ways, foiled attempts at the expansion of human understanding.

So, back to the thickness or height,  $h$ , of fluid drops: if we hold our  $2\text{-kg}$  brick above the floor, it exerts a downward force on the hand that holds it. This force is its weight,  $2\text{kg}$  of force. While we hold it, its downward speed, with respect to the earth, is zero. If we drop it, its downward speed increases from zero until it hits the floor. It accelerates as it drops. The acceleration,  $g$ , is the acceleration due to gravitational attraction of the earth or, better, the mutual attraction of brick and earth: the earth, being much bigger, does not fall very far toward the brick! If bricks don’t inspire you, use Newton’s apple! It falls with the same acceleration as a brick.

The downward force,  $F$ , on the brick is the product of its mass,  $M$ , and the acceleration,  $g$ , due to gravitational attraction.  $F = M \cdot g = W$  is the weight of the brick on earth. Similarly, the downward force on a

puddle of water or mercury is the weight of the puddle, or its mass,  $M$ , times the acceleration,  $g$ , due to gravitational attraction between the puddle and earth. We now have established that the dimensions of force  $F$ , are:

$$[\text{mass}] \cdot [\text{acceleration}] = [F] = \left[ \frac{M \cdot L}{T^2} \right] \quad (2)$$

Acceleration  $g$  has dimensions  $[L/T^2]$ ; distance (km) per hour per second. Surface tension  $\gamma$  has dimensions  $[M/T^2]$ —mass per hour per second?

Surface tension  $\gamma$  is best described as the energy that keeps the droplet of mercury or water from collapsing. This energy accomplishes this by forming a sort of “skin” on the surface of the droplet, or puddle. It is “energy per unit surface”. We shall, therefore, have to investigate the dimensions of energy. Energy, as everyone who works for a living knows, is the ability to do work! It is the energy to do something. Our  $2\text{-kg}$  brick, held in hand, has energy! If you drop it on your toe it will do work! The energy  $E$  in the brick in hand, available for doing work on your toe, is the product of its weight (the force  $F$  it exerts on your hand) and the distance  $d$  it falls before it hits your toe. The dimensions of energy are force time distance.

We have found that force  $F$  is mass  $M$  times acceleration and acceleration  $g$  is distance per time per time. Thus, the dimensions of energy are:

$$[E] = [F \cdot d] = [M \cdot g \cdot d] = \left[ M \cdot \frac{L}{T^2} \cdot L \right] = \left[ \frac{M \cdot L^2}{T^2} \right] \quad (3)$$

In passing, we may remark that the dimensions of velocity,  $v$ , are  $[L/T]$ , distance per unit time. Thus

$$[E] = [M \cdot v^2] \quad (4)$$

This fits nicely with Einstein’s deduction that  $E = M \cdot c^2$ , therefore we must be on the right track.

We may now discover the dimensions of surface tension,  $\gamma$ :

$$[\gamma] = \left[ \frac{\text{energy}}{\text{area}} \right] = \left[ \frac{E}{L^2} \right] = \left[ \frac{M \cdot v^2}{L^2} \right] = \left[ \frac{M \cdot L^2}{T^2} \right] = \left[ \frac{M}{T^2} \right] \quad (5)$$

Finally, we may find the height,  $h$ , of the drops and puddles. What sort of function shall this be? Shall it be an exponential function?

$$h = f(\rho, \gamma, g) = c \cdot d^x \cdot \gamma^y \cdot g^z \quad (6)$$

where  $c$  is a number to be determined.

Dimensionally,

$$[L] = \left[ \frac{M}{L^3} \right]^x \cdot \left[ \frac{M}{T^2} \right]^y \cdot \left[ \frac{L}{T^2} \right]^z = [M^0 \cdot L^1 \cdot T^0] \quad (7)$$

Since we are after  $[L]$  it has an exponent 1 and  $[M]$  and  $[T]$  have exponents zero.

Dimensions on both sides of the equal sign must be the same, so

$$\begin{aligned} x+y &= 0 \\ -3x+z &= 1 \\ -2y-2z &= 0 \end{aligned}$$

then

$$\begin{aligned} x &= -y \\ 3x &= z-1 \\ z &= -y \end{aligned}$$

then

$$\begin{aligned} x &= z \\ 3x &= x-1 \\ z &= x \end{aligned}$$

then

$$x = -1/2; y = 1/2; z = -1/2$$

and

$$h = c \times \rho^{-1/2} \times \gamma^{1/2} \times g^{-1/2} \quad (8)$$

or

$$h = c \sqrt{\frac{\gamma}{\rho \cdot g}} \quad (9)$$

Let us now find if this formula actually works!

Look up the values for  $\rho$ ,  $\gamma$  and  $g$  in a handbook and we find the characteristics shown in Table 1.

We also find acceleration due to gravity,  $g = 9.80$  m per second per second.

Since the numbers we looked up are all in the metres, kilogram and second system ( $L$  in m,  $M$  in kilograms,  $T$  in seconds), the answers we calculate will appear in metres.

$$\text{For mercury, } h = c \sqrt{\frac{0.48548}{(13500)(9.80)}} = 0.00192c$$

$$\text{For water, } h = c \sqrt{\frac{0.07423}{(1000)(9.80)}} = 0.00275c$$

We did not determine the dimensionless constant  $c$ , but if you watch the water beads on your newly waxed car, I feel sure you can decide that  $c$  is very close to 1. In other dimensional exercises, even very complicated ones, we find that *Nature* likes

**Table 1.** Density and surface tension for mercury and water.

Physical quantity	Mercury	Water	Units
Density, $\rho$	13,500	1000	$\text{kg m}^{-3}$
Surface tension, $\gamma$ (25°C)	0.48548	0.07423	N/m (N/m) = $\text{kg} \cdot \text{s}^{-2}$

her constants to be simple, like 1, 2,  $\pi$ ,  $3\pi/4$  etc.

### Eliminating the necessity of time and mass units of their own

Currently, the International Standard system of dimensions and units (SI) is summarised as follows in the 2011–2012 *CRC Handbook of Chemistry and Physics*: “The core of the *SI* is the seven base units for the physical quantities length, mass, time, electric current, thermodynamic temperature, amount of substance, and luminous intensity”. You may already know all this, but I feel its statement is needed, because it contains at least one important false implication! It also implies that there is something fundamental about the seven base units. You may reply, “So what? We all know that the metre is a fraction of the earth’s circumference, the second 1/86400 of a solar day, the kilogram the weight of a litre of water, etc. ...”

We now have better ways of measuring the metre, kilogram and second! We also measure mass in electron volts, length in terms of reproducible wave lengths, time using vibrating crystals, and so on.

Before proceeding, may I repeat the dimensional analysis that discovers the height,  $h$ , of beads of liquid on a smooth surface, using a slightly different approach.

We found the dimensions of density:

$$[\rho] = \left[ \frac{M}{L^3} \right] \quad (10)$$

This gives mass the dimensions:

$$M = [\rho \cdot L^3] \quad (11)$$

We found the dimensions of force:

$$[F] = \left[ \frac{M \cdot L}{T^2} \right] \quad (12)$$

Pressure,  $P$ , is force per unit area:

$$[P] = \left[ \frac{M \cdot L}{L^2 \cdot T^2} \right] = \left[ \frac{M}{L \cdot T^2} \right] \quad (13)$$

If we put (11) in (13):

$$[P] = \left[ \frac{\rho \cdot L^3}{L \cdot T^2} \right] = \left[ \frac{\rho \cdot L^2}{T^2} \right] \quad (14)$$

From this, dimensions of time are given by:

$$[T^2] = \left[ \frac{\rho \cdot L^2}{P} \right] \quad (15)$$

We have now invented a new dimensional system,  $LP\rho$ , replacing the conventional  $LMT$  (i.e., length, mass, time) part of the *SI* system, in which there is no need for separate dimensions for  $[M]$  and  $[T]$ .

Returning to the problem of the height of beads of liquids:

$$h = f(\rho, \gamma, g) = c \cdot \rho^x \cdot \gamma^y \cdot g^z \quad (16)$$

Dimension of density:

$$\left[ \frac{M}{L^3} \right] = [\rho] \quad (17)$$

Dimension of surface tension:

$$\left[ \frac{M}{T^2} \right] = \left[ \frac{\rho \cdot L^3 \cdot P}{\rho \cdot L^2} \right] = [P \cdot L] \quad (18)$$

Dimension of acceleration:

$$\left[ \frac{L}{T^2} \right] = \left[ \frac{L \cdot P}{\rho \cdot L^2} \right] = \left[ \frac{P}{\rho \cdot L} \right] \quad (19)$$

Dimensionally:

$$[L]^1 = [\rho]^x \cdot [P \cdot L]^y \cdot \left[ \frac{P}{\rho \cdot L} \right]^z \quad (20)$$

For dimensional homogeneity:

$$\begin{aligned} x-z &= 0 \\ y+z &= 0 \\ y-z &= 1 \end{aligned} \quad (21)$$

As before:

$$x = -\frac{1}{2} \quad y = \frac{1}{2} \quad z = -\frac{1}{2} \quad (22)$$

continued on page 11

# Improved Food and Feed Safety through Systematic Planning and the Theory of Sampling (TOS): An Introduction to “GOODSamples”

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The Food Safety Modernization Act (FSMA), signed into law by President Barack Obama on 4 January 2011, provides the US Food and Drug Administration (FDA) with a framework to better protect public health by strengthening the food safety system. Its primary purpose is to ensure the safety of the US food supply by shifting focus to prevention of food and animal feed contamination through enhanced partnerships and integration among federal, state, local, tribal and territorial partners. FSMA is the most sweeping reform of US food safety laws in 70 years. FSMA addresses Preventive Controls, Inspection and Compliance, Response, Imported Food Safety, and Enhanced Partnerships. Title II—Improving Capacity to Detect and Respond to Food Safety Problems—addresses Laboratory Accreditation for Analyses of Feeds in Section 202. Section 202(a)(6) states that Model Standards will require appropriate sampling.

The US FDA awarded a five-year cooperative agreement to the Association of Public Health Laboratories (APHL), Association of Food and Drug Officials (AFDO) and the Association of American Feed Control Officials (AAFCO) to support the implementation of The Food Safety Modernization Act (FSMA). One of the Specific Aims in the cooperative agreement is “Harmonized Policies and Procedures for Equivalency of Data”. A task under this Aim is to establish a working group to develop harmonised policies and procedures for sample collection, shipment, analysis, storage and retention of food and feed materials. The *Sampling and Sample Handling Working Group* effort is led by AAFCO due to its long history of recognition of sampling and sample preparation as critical aspects of the regulatory process.

Currently, procedures for sample collection are as varied as the number of agencies that collect samples. This wide variety of sample collection techniques does not lend itself to data equivalency among the various agencies, a prerequisite for inter-agency data sharing because of uncontrolled sampling bias (which cannot be corrected) and other sampling errors (see further below). The goal of the working group is to develop a common sampling strategy for sampling food and feed. With this common sampling strategy, data can be evaluated with respect to “fit for purpose” or, more aptly, “fit for decision” criteria for any agency, project or situation. This will allow for harmonised data collection, defensibility of

analytical results and, ultimately, the ability of agencies to share data with confidence. The main audience for this document is regulatory programmes and their associated laboratories, including management, inspectors, quality assurance officers and laboratory personnel.

The guidance document currently under construction has been titled *Guidance on Obtaining Defensible Samples or GOODSamples*.

## GOODSamples, the philosophy

The common perception is that all that is needed are more standard operating procedures (SOP), but as there are already thousands available; it is doubtful that “just a few more” will meet the goals of FSMA. Unfortunately, there are an infinite number of sampling scenarios. SOPs are stagnant and not responsive to new analytes, new regulations, new sampling techniques and tools, unanticipated field conditions, new field analytical techniques etc. But nowhere has the characteristic “representative” been given a full, operative definition by the US FDA or food/feed regulatory bodies. Therefore, the philosophy of *GOODSamples* is not to develop more Standard Operating Procedures for sample collection, but to provide a practical and complete framework for field inspectors, project managers, chemists etc. to work together to develop and implement sampling protocols to meet the objectives of FSMA. This can only be met by insisting on documented representative sampling procedures through the entire field-to-aliquot pathway (see Figure 1).

The FDA developed many sampling protocols based on attribute sampling strategies ca WWII that predated the Theory of Sampling (TOS). The scientific basis for these protocols has changed little over time. FSMA has now brought an opportunity to change and update the science behind sampling food and feed.

## GOODSamples, the document

It should be evident that data equivalency is dependent upon the collection of representative samples for specific objectives; it may not be so readily evident that representative sampling is a function of the whole organisation and a management system that is committed and promotes communication.

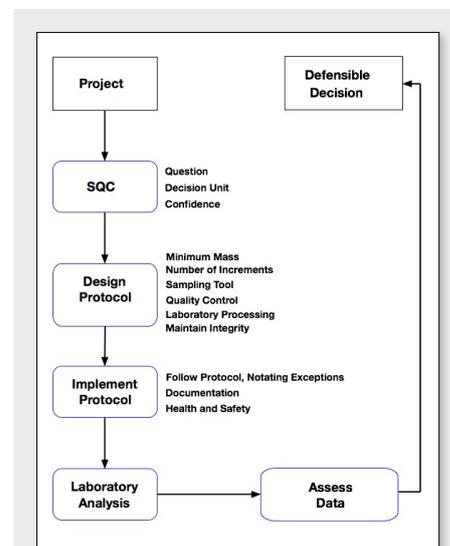


Figure 1. GOODSamples pathway to a defensible decision

*GOODSamples* address the entire process of sampling from development of objectives to final assessment. Communication between all disciplines involved in sample collection is stressed throughout the document. The specific chapter titles and the rationale for its inclusion are as follows:

*Definitions:* A common vocabulary is essential! Each segment of the intended audience currently has different terms for the same concept.

*Management Considerations:* Supportive and knowledgeable management is critical to a successful sampling programme. Communication among management, quality, sampling and laboratory staff is needed to develop competent sampling protocols. This chapter provides a rationale for the importance of management in the overall effort.

*Sampling Quality Criteria (SQC):* SQC provides the framework for planning and managing practical sampling and analytical operations consistent with the food/feed programme needs. It is a series of statements that clarify technical and quality criteria to support defensible decisions. This chapter introduces the key elements of SQC.

*Theory of Sampling (TOS):* The most important part of sample collection is a basic understanding of the TOS and what makes a representative sample. Understanding TOS is key for management, quality assurance staff, inspectors, laboratory analysts and data users. Only a brief introduction to the topic is presented here; the standard *DS 3077—Horizontal*<sup>1</sup> gives additional background information and references and Gy<sup>2</sup> and Pitard<sup>3</sup> provide greater detail on the TOS.

*Quality Control:* This chapter describes the three of the four general types of quality control checks used in the sampling process. These include checks for contamination from various sources, sampling repeatability (precision) and laboratory consistency.

*Sampling Tools:* This chapter is divided into two sections: *The Theory of Sampling Tools* and *Sampling Tools*. The first section addresses the theory of the selection of equiprobable particles, sample correctness and the dimensions of decision units. The second section addresses considerations in choosing the correct tool for a specific sampling effort.

*Maintaining Integrity* This chapter is divided into sections on *Evidentiary Integrity* and

*Analyte Integrity.* Evidentiary integrity is maintained by thorough documentation, including chain of custody. *Analyte Integrity* is maintained by proper preservation, proper choice of containers, observance of holding times and proper handling, packaging and shipping.

*Health and Safety:* SAFETY FIRST! No sample is as important as your safety.

*Sampling Protocol Design:* A sampling protocol is a detailed procedure for obtaining a representative primary sample of appropriate mass and number of increments from a specific decision unit to meet the SQC. The protocol includes the appropriate quality control and directions for maintaining evidentiary and analyte integrity, tool usage, sample processing etc.

*Examples of Sampling Protocols:* Examples of protocols for a specific SQC are provided.

*Laboratory Sampling, Handling and Preparation:* This section provides guidance on how laboratories should handle and process samples received for analysis, keeping in mind two primary responsibilities: ensuring that the target analyte(s) are not compromised during sample preparation and storage; and obtaining representative analytical samples and test portions from the laboratory sample.

*Process Assessment:* Assessment of the entire process is critical to determine whether it meets the objectives set forth (SCQ) and is suitable to make decisions at the specified confidence.

## GOODSamples, the approach

The approach promulgated in *GOODSamples* is that all sampling protocols must begin with development of appropriate objectives. Too often, data is generated without objectives first being defined. A lack of objectives, or poorly defined objectives, unavoidably leads to undesirable outcomes. These include inconsistency in interpretation of results; questions are not answered directly; insufficient confidence; and/or inefficient allocation of resources. As Bernard Baruch has stated, "A problem well stated is a problem half solved."

Sample Quality Criteria (SQC) provides the framework to determine project objectives and is the basis of design for a sampling protocol to answer a specific question with a known confidence (see Figure 2). Once the SQC is established, the sampling protocol can be developed based on TOS incorporating necessary quality control.

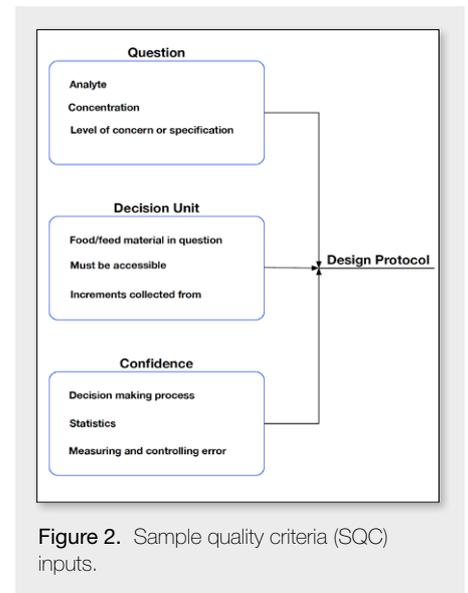


Figure 2. Sample quality criteria (SQC) inputs.

Care must be taken to ensure the analyte integrity is maintained through the entire process including transportation to the laboratory. Laboratory sub-sampling and processing protocols need to be considered and included as part of the protocol. The *AAFCO Guidelines for Preparing Laboratory Samples*<sup>4</sup> provides guidance to feed laboratories with basis in TOS and was used as a basis for International Standard 6498:2012 *Animal feeding stuffs—Guidelines for Sample Preparation*.<sup>5</sup> Both will serve as normative references for *GOODSamples*. Petersen *et al.*<sup>6</sup> also provide a TOS-approach to laboratory processes for dry granular feed materials.

There must be established a unified responsibility (institutionally, through normative good practice documents). Three primary elements of SQC in *GOODSamples* are:

### What is the question sampling and analysis is intended to answer?

Identification of the analyte(s) and concentration level(s) of concern is the first consideration in SQC. It is critical that this is known in advance so planning ensures that appropriate sample containers are used, sampling tools and techniques can maintain the integrity of the analyte(s) are utilised, analytes are preserved appropriately and health and safety is addressed.

Determination of the expected analyte concentration of concern is also important in the development of the sampling protocol. If the concentration is unknown and a reasonable estimate is not available,

a specification limit may be used as the concentration estimate since this is the concentration where the error must be closely controlled.

In situations where there are multiple analytes of concern, this information is required for all analytes.

**What is the decision unit (population, lot) the sample is intended to represent?**

For some scenarios this is an obvious and easy question to answer, but in reality, identification of the decision unit is typically not considered. “Just take some samples” is a typical approach. This aspect of sampling can be the most difficult to understand initially, but it is the most fundamental aspect of sampling. The decision unit determines what needs to be accessible; where increments are collected from, where inferences are made to, which tools will select the right shape and mass of increments. It is critical that the entire decision unit be available to the sampler; this is termed the fundamental sampling principle (FSP) in TOS.

**What is the desired confidence in the final decision?**

Selecting the level of confidence can be difficult for those without some level of statistical understanding, especially if a specific number on the level of confidence (e.g. 95%, 99%) is desired. Confidence is actually a function of consequences. The more serious the consequences of the ultimate decision, the greater the level of confidence needed. Confidence does not have to be statistical, but it does have to be agreed on by all the parties involved.

Knowing how the data is going to be applied is critical to ensure that the appropriate data is collected. An often-overlooked aspect in the planning stage is to specify how the data will be applied in making the decision. This may include the number of samples, types of sampling, allowable sampling error, quality control, sample processing, analytical methods and a host of other important design aspects. All too often, the intended decision cannot be made because the data are inadequate for the type of decisions required by the SQC.

Quality control is an important, yet often overlooked, element in the confidence realm. Quality control demonstrates that the system is in control and allows an empirical estimation of the effective, total sampling

and analysis error. One type of quality control is a control for the detection of contamination. The contamination may be from the environment, tools or containers. This is important for sampling of trace, volatile or biological analytes. Replication (in the form of a “replication experiment”, DS 3077 82013) is another approach that can be used to determine the total measurement uncertainty (MU) [sampling + analysis] associated with the analytical results. Esbensen and Wagner<sup>7</sup> outlined the complementary, interacting competences between TOS<sub>sampling</sub> and MU<sub>analysis</sub>.

Once the SQC process is complete, the design of the sampling protocol can begin. The sampling protocol is impossible to develop without a competent understanding of TOS. To the knowledge of the authors, TOS has never been comprehensively included in food and feed SOPs in the United States.

Once the sampling protocol has been designed, implementation can begin. Unfortunately, field situations are seldom what were anticipated during the development of the sampling protocol and sometimes adjustments must be made. If the sampler is following a protocol blindly, unrepresentative samples may be the result. The ultimate data user is typically unaware of the field conditions and makes

decisions based on results from samples that may not be adequate for the objectives of the project. Therefore, it is of paramount importance that the people collecting the primary samples have sufficient training in all aspects of SQC and TOS so they can adapt to unanticipated conditions in the field without compromising the integrity of the primary samples and the resulting decision. Training is an important part of FSMA and critical to the implementation of *GOODS*amples.

Often the data is used without any determination or assessment as to whether it meets the objectives set forth and is suitable to make decisions. Assessment of data includes evaluation of the appropriateness of the SQC, critical review of the quality control data (not just pass/fail), error propagation calculations, verification of data assumptions if statistical calculations are performed etc. In other words, did everything go as planned? If not, what impact does that have on the confidence in the final decision? (see Figure 3).

**Summary**

Sampling is more than a collection of Standard Operating Procedures that are selected for ease of use or availability of equipment. Simply filling containers will not

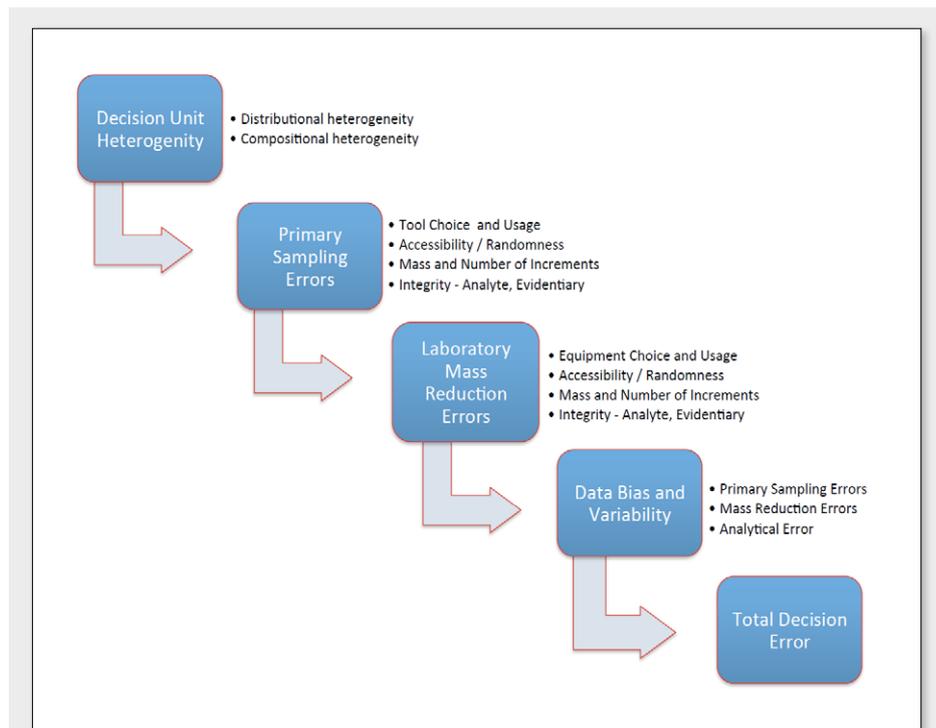


Figure 3. Contributions to the Total Decision Error.

provide useful data for defensible decision-making. Sampling is about meeting project objectives. A systematic approach to meeting project objectives is the most critical, but largely overlooked, part of the process. *GOODSamples* provides the necessary and sufficient framework to allow for defensible decisions.

The guidance provided in *GOODSamples* is not unique to food and feed sampling but should be applicable to any sampling effort. The process from determining objectives to a developing a final sampling protocol must be based on science. While there are many proposals for practical approaches to sampling, mostly of a highly specific, “home-grown” variety, from which no general conclusions could possibly be drawn, TOS reigns as the most comprehensive approach for the types of materials encountered in the food and feed industries. In the recently codified form, *DS 3077*<sup>1</sup> (2013) will be a normative reference for *GOODSamples*. While the focus of *GOODSamples* is food and feed regulatory programmes and their associated laboratories, the document will also be suited for producers, distributors and manufacturers of food and feed. Other industries such a fertiliser, pharmaceutical and supplement producers can also benefit from a systematic approach such as outlined in *GOODSamples*.

Many readers of the *TOS Forum* have experience with the sampling of food, feed, fertiliser, pharmaceuticals, supplements

and other related commodities. The present authors seek relevant references to cite in the new guidance document. We wish to include as much international “flavour” as possible, since most of the work in TOS does not take place in the United States. Please feel welcome to contact the corresponding author with any comments, questions, issues, concerns and references you have.

### Acknowledgements

This publication was supported by the Association of Public Health Laboratories (APHL) and by Grant No. 1U18FD004710 from the Food and Drug Administration (FDA) of the US Department of Health and Human Services. Its contents are solely the responsibility of the authors and do not necessarily represent the official views of APHL or FDA.

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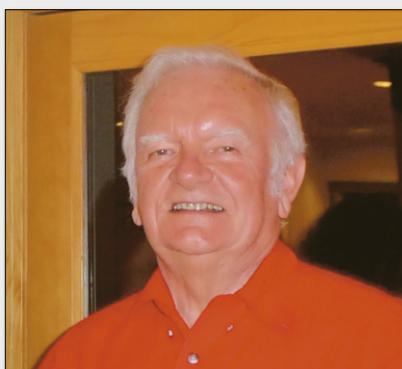
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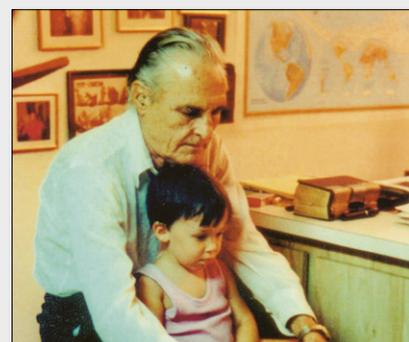
$$h = c \sqrt{\frac{\gamma}{\rho \cdot g}} \quad (23)$$

At this point, this only shows the obvious, that the  $LP\rho$  and  $SI$  systems are equally useful in resolving this small problem. It also shows that there is nothing sacrosanct about length, mass and time as primary dimensions.

The importance of this lesson, a simplistic appetiser, does not fully appear until the other “dimensionally independent base quantities”, the thermal and more especially the electromagnetic quantities are taken into consideration, which have huge implications; but this will be addressed in subsequent publications.



**Francis Pitard**, gold medal recipient from the World Conference on Sampling and Blending, combines his experience in nuclear chemistry, analytical chemistry, geochemistry, and statistical process control in consulting and auditing of many international companies and teaching short courses on sampling. He lives in Colorado, USA.



**Charles Oliver Ingamells (1917–1994)**  
A simpler system of dimensions and units  
“The International System of Units adapted “seven dimensionally independent quantities” that are measured with their respective units. These quantities are not independent and some of them do not deserve their own units. Therefore, it becomes conceivable that our ways of thinking today in Physics and Astronomy are flawed, or at least unnecessarily complicated”.

# Agreement analysis—testing the boundaries between producers and consumers

Maxime Pitard  
HonuaTek LLC

## Introduction

The most serious mistakes are not being made as a result of wrong answers. The truly dangerous thing is asking the wrong questions. These words of wisdom from the late Peter Drucker underscore the importance of being well informed when making business decisions. This is especially true of producer–consumer boundaries in industrial processes, where the quality of a lot, or series of lots, can have significant financial consequences.

In order to mitigate any disagreement at these boundaries, concerned parties typically establish common protocols to monitor the quality of the lots produced. These monitoring protocols—often implemented with agents from a combination of consumers, producers and/or commonly agreed-to third parties—consist of splitting samples taken from the lots of interest and processing them separately

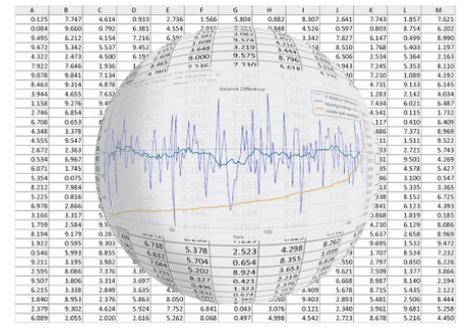
to generate two, or more, sets of estimates which are then compared for differences (Figure 1).

These monitoring protocols typically define:

- the party(ies) responsible for taking the samples;
- the party(ies) responsible for processing and analysing the samples;
- threshold(s) for disagreement between the estimates;
- the steps to take when estimates disagree.

This last point is of particular interest, as many organisations will make use of an umpire sample (also known as a referee or reserve sample) as the arbiter of any disagreement found in the estimates. This sample is often kept to the side, and used on an as-needed basis to resolve contractual disputes as per the protocol that defines its use. Regrettably, if the goal is to infer a deeper understanding of the source of disagreement and take corrective action on the offending process, then using the umpire sample for this purpose may very well provide the right answer to the wrong question.

Pierre Gy, in his ground-breaking book on the Theory of Sampling (TOS), dedicated two chapters to the problem of producer–consumer boundaries, where he presented an approach for testing for agreement between two series of independent estimates of a sample characteristic.<sup>1</sup> In these chapters he cautioned his readers on how typical bias testing is more focused on controlling risk from a seller’s point of view, at an unknown and potentially significant risk to the buyers. He provided guidance in addressing this issue, and developed a simple and systematic approach to help monitor for the presence of statistically significant bias in a process: what we call the Gy bias test, or continuous bias test<sup>2</sup> (for its control chart-like properties).



FPSC Sampling Consultants in collaboration with HonuaTek saw the value in Pierre Gy’s approach and built a software package to automate its use. As the software was developed and used with internal clients, it became clear that Gy’s approach, although powerful, did not provide a complete view of the processes that generated the estimates; it was not enough to visualise the progression of bias over time, we needed to better understand the possible sources of this bias as well. Building upon the work of Pierre Gy and others,<sup>3</sup> the software was extended with additional capabilities to provide a more complete set of tools.

The resulting “Agreement Analysis” software incorporates a complementary set of techniques, originating from the fields of statistical process control and sampling statistics. It enables users to study the same data through different lenses, providing as complete a view as possible, while keeping the complexity down to a minimum.

Agreement Analysis currently supports five techniques (Figure 2):

- the Gy Bias Test to identify statistically significant bias between the estimates;
- the Scatter Plot to examine correlation between the estimates;
- the Relative Difference plot to investigate differences between estimates and their potential source;

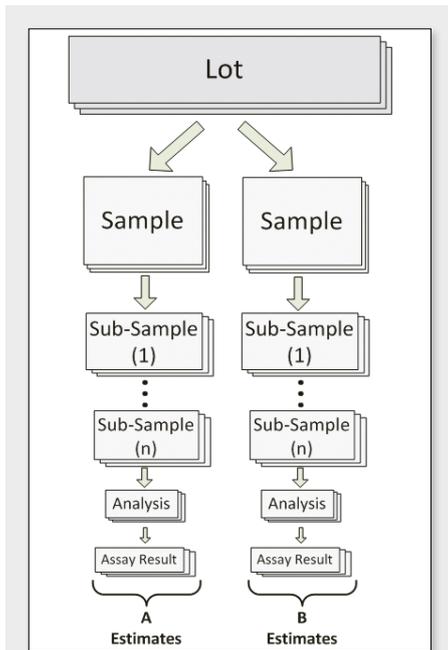


Figure 1. Lot estimation.

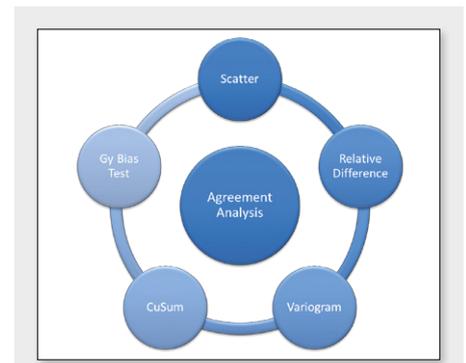


Figure 2. Five techniques.

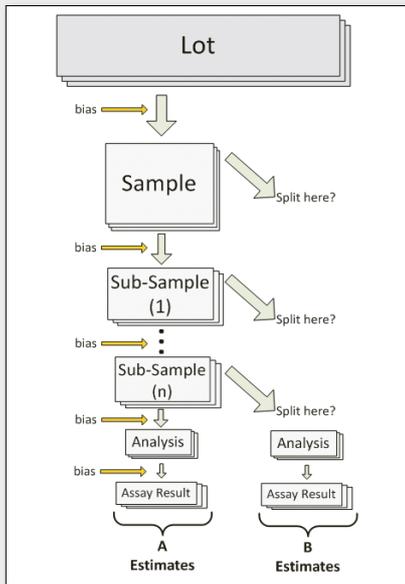


Figure 3. Source of bias.

- the Cumulative Sum plot to compare short-term trends in the estimates;
- the Variogram to compare the variability of the estimates over time.

Should the estimates be found to disagree, results from the different techniques can be combined to provide an overall synthesis of the possible sources of the disagreement.

By seamlessly integrating these five techniques, the Agreement Analysis software provides a collection of tools to analyse differences between sets of estimates, helping those responsible for quality control focus on answering the following key questions:

- Is the level of risk taken by both producer and consumer acceptable?
- Are the differences between the sets of estimates tolerable?
- If the estimates disagree, which set is most likely incorrect and why?

### Background

TOS shows us that bias generating errors are present at every step where samples are handled in a lot estimation protocol—the introduction of bias cannot be avoided. The best that can be done is to minimise the amount of bias introduced at every step by following appropriate sampling practices.

There are a several stages in a lot estimation protocol where a sample can be split to generate the two sets of estimates needed for analysis (Figure 3). It is important to understand that any bias introduced prior to the sample split will be present in

both sets of estimates and will be difficult to differentiate as coming from the protocol or the lot itself. To this end, the sooner the sample is split in the overall protocol, the more information is available to identify the sources of bias. This of course has to be weighed against the added cost of carrying out the full protocol on each sample.

Once the estimates are available, getting them into the Agreement Analysis software and applying the five techniques described in the following sections is a straight forward process.

### Scatter

The scatter plot is a good starting point for analysis since this technique presents a simple visual indicator of how well one set of estimates can predict the values of the second set of estimates (Figure 4).<sup>4</sup>

This approach tests one of the main assumptions for agreement analysis; that the two sets of estimates are good linear predictors of each other, given they are different estimates of the same initial samples or sub-samples.

The plot consists of pairing up the estimates and plotting them on the graph, with the expectation that these will form a narrow dispersion ellipse along the identity line  $x=y$ . The plot can be inspected for a linear relationship, and the transverse of the dispersion ellipse estimated by a best fit line calculated using least-squares linear regression.

How well the estimates agree can be in-part measured by the correlation coefficient. Any random error introduced in the estimation protocol has the effect of widening the ellipse. Any systematic error in the estimates, depending on its nature, can

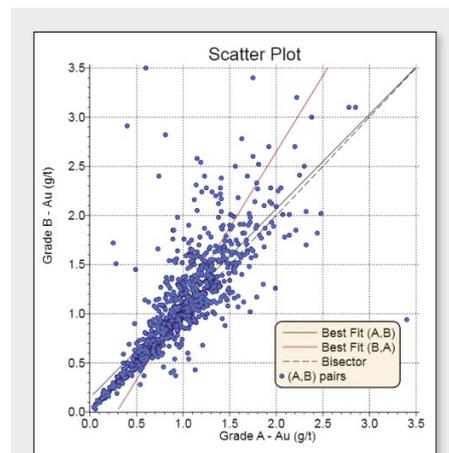


Figure 4. Scatter plot.

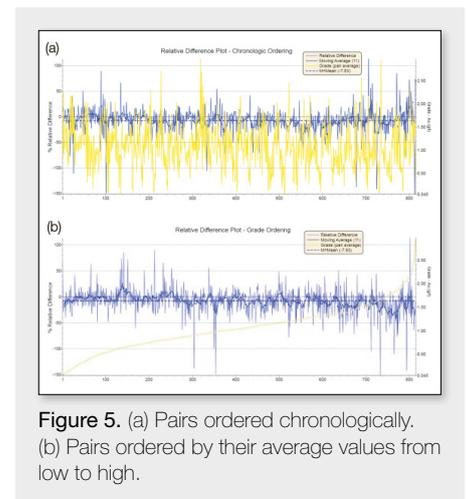


Figure 5. (a) Pairs ordered chronologically. (b) Pairs ordered by their average values from low to high.

shift the transverse of the ellipse away from the identity line as well as change its slope.

### Relative difference

The second technique used in the analysis is the relative difference plot, which provides a convenient control chart focused on detecting bias between the sets of estimates. This technique relies on several key plots: the average of the estimates, the per cent relative difference for each pair of estimates, the moving average of the per cent relative difference. This technique supports ordering the pairs of estimates in one of two ways:

- Pairs ordered chronologically; useful in detecting problems such as slow drift in one of the estimated sets (Figure 5a).
- Pairs ordered by their average values from low to high; providing information on how the differences fluctuate with the change in the estimates (Figure 5b).

### Variogram

Adapting an approach introduced by Pentti Minkkinen,<sup>3</sup> the variographic technique is utilised to compare the process variability between the sets of estimates.

$$V_j = \frac{1}{2(N-j)} \sum_{i=1}^{N-j} [h_{i+j} - h_i]^2, j = 1, 2, \dots, \frac{N}{2}$$

The absolute variogram ( $V$ ) of each set of estimates is calculated for the data increments ( $h$ ) and lag ( $j$ ), which are then compared for differences.

Items of particular interest for comparison from the variographic study include the short range, long range and cyclic terms,<sup>5</sup> (Figures 6a and 6b) which can also be used as input to other techniques such as the cumulative sum plot and Gy's bias test.

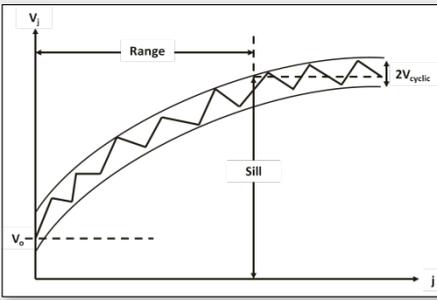


Figure 6a. Variogram.

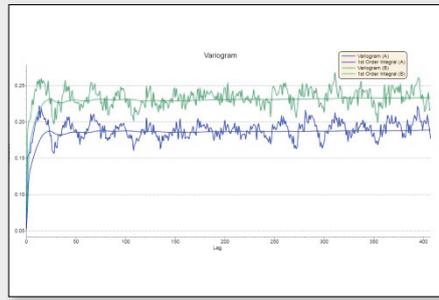


Figure 6b. Comparing variograms.

### Cumulative sum

The cumulative sum plot is a tool commonly used in statistical process control to detect small changes in mean level.<sup>6</sup>

For agreement analysis, a variant of this plot, the Tabular Cusum,<sup>7</sup> has been adapted to create control charts to compare the effect of the accumulation of small, persistent, non-random changes between the sets of estimates (Figure 7). This technique requires identifying both a target value ( $\mu_0$ ) separating the plot into upper and lower portions; and a slack value ( $K$ ) to filter out small random fluctuations. The upper portion of the cusum ( $C^+$ ) shows the cumulative effect of positive deviations from the target value with a minimum possible value of zero, and is defined by the recursive function:

$$C_0^+ = 0$$

$$C_i^+ = \max[0, C_{i-1}^+ + x_i - (\mu_0 + K)]$$

The lower portion of the cusum ( $C^-$ ) shows the cumulative effect of negative deviations from the target value with a maximum possible value of zero, and is defined by the recursive function:

$$C_0^- = 0$$

$$C_i^- = \max[0, C_{i-1}^- - x_i + (\mu_0 - K)]$$

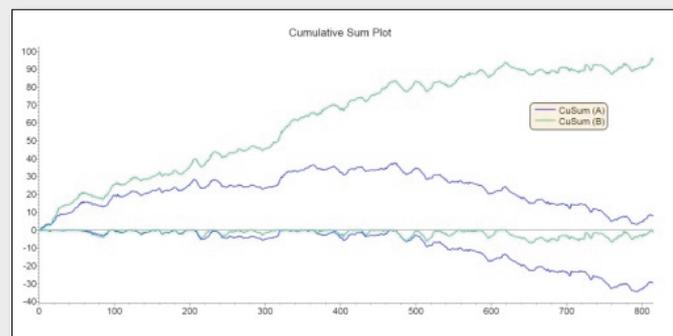


Figure 7. Cumulative sum plot.

Typically, the construction of the cumulative sum plot requires a statistical model derived from historical data to establish values for parameters such as  $K$ . This is of lesser concern when applied to agreement analysis since the focus is not on identifying out of control situations, but instead on comparing the two sets of estimates.

### Pierre Gy's Bias Test

This adaptation of classic hypothesis testing is used as the final technique of agreement analysis, and helps establish the statistical significance of observed differences.

Based on Pierre Gy's approach, this technique helps users visualise the evolution of bias over time when comparing two sets of estimates (Figure 8). The test involves taking each pair of estimates, and given a certain risk  $\alpha$ , defining a random variable for a preliminary test  $W$  (testing for no difference in the pairs),

$$W_i = \frac{D_i \sqrt{i}}{s_i}, i = 1, 2, \dots, N$$

and one or more random variables for the complementary tests  $W'$  (testing for a tolerated systematic difference in the pairs).

$$W'_i = \frac{(|D_i| - D_a) \sqrt{i}}{s_i}, i = 1, 2, \dots, N$$

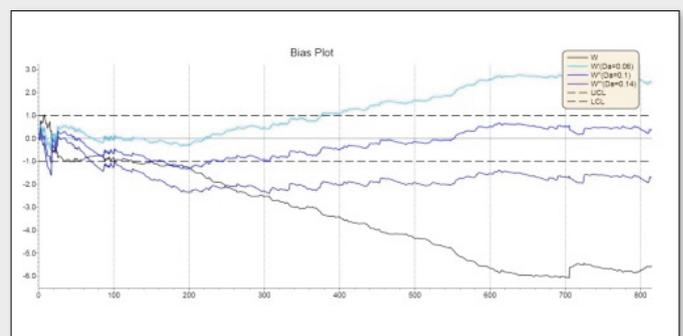


Figure 8. Bias plot.

Where  $D_i$  is the estimate of the systematic differences,  $s_i$  the estimate of the standard deviation of the systematic differences and  $D_a$  the tolerated systematic difference.

Normalising the results of the tests over the t-distribution, we are able to construct an easy to interpret control chart with statistically significant conclusions between the random variable values of  $-1$  and  $+1$ .

The control chart allows for a rich set of interpretations described in detail in Table 1, where  $W$  and  $W'$  are the results of the preliminary and secondary tests respectively.

### Software features

Designed for ease of use, the Agreement Analysis software features a rich set of capabilities, including the ability to create an unlimited number of views into the data. Each view can be constructed from one of the five supported techniques and then uniquely configured for a user's specific requirements.

Comparative analyses can be performed by seamlessly transitioning amongst the different views, which can in turn be stored, along with the data, to project files for future retrieval.

With the ability to import data from various sources such as Microsoft Excel, the software provides complete customisation (styles, sizes and colours) of the displayed graphics and fonts (Figure 9).

Users have the option to save the graphics from the views to JPEG format, and reports containing all the views, along with their high resolution graphics and configuration parameters, can be generated and exported to Microsoft Word. These reports are stand-alone documents which can be fully edited by the users.

FPSC and HonuaTek will continue to collaborate and improve upon the capabilities of the Agreement Analysis software, driven

Table 1.

$W$	$W'$	Interpretation
$>+1$	$>+1$	With the given risk we can conclude that set A is systematically higher than set B, and beyond the tolerated systematic difference.
$>+1$	$[-1,+1]$	With the given risk we can conclude that set A is systematically higher than set B—but we cannot determine if it is beyond the tolerated systematic difference.
$>+1$	$<-1$	With the given risk we can conclude that set A is systematically higher than set B, but within the tolerated systematic difference.
$<-1$	$>+1$	With the given risk we can conclude that set A is systematically lower than set B, and beyond the tolerated systematic difference.
$<-1$	$[-1,+1]$	With the given risk we can conclude that set A is systematically lower than set B—but we cannot determine if it is beyond the tolerated systematic difference.
$<-1$	$<-1$	With the given risk we can conclude that set A is systematically lower than set B, but within the tolerated systematic difference.
$[-1,+1]$	$[-1,+1]$	With the given risk, we do not have enough samples to draw a conclusion.
$[-1,+1]$	$<-1$	With the given risk we cannot conclude the presence of a systematic difference between set A and set B—if it does exist, it is less than the tolerated systematic difference.
$[-1,+1]$	$>+1$	The results are erroneous and no conclusion can be drawn from them.

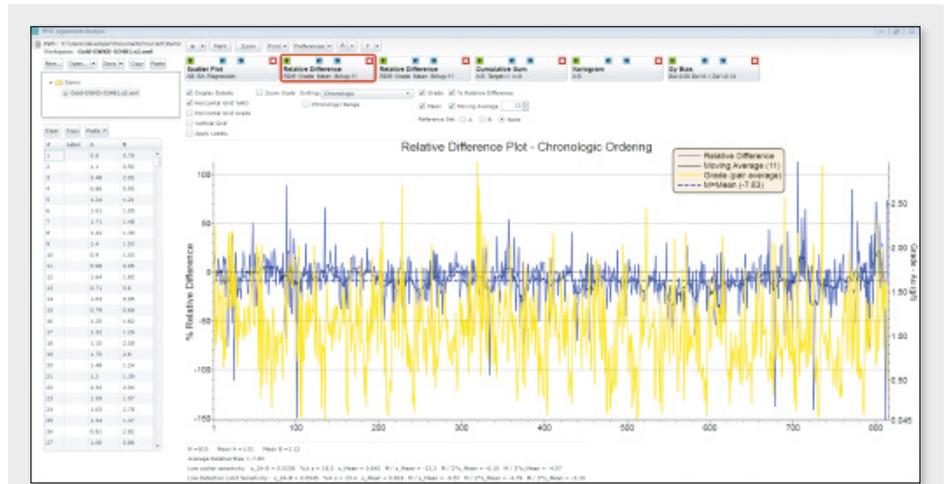


Figure 9. Software.

by experience and continuous feedback from our growing user community—helping provide the right answers to the right questions.

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# A critical assessment of the HGCA grain sampling guide

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HGCA's grain sampling guide is assessed with respect to the principles for representative sampling as set forward in the Theory of Sampling (TOS). Sampling correctness, which requires the elimination of all Incorrect Sampling Errors (ISE), constitutes the only guarantee for valid, representative grain quality control; presence of ISEs causes a varying, uncontrollable sampling bias that cannot be corrected for. Contrary to a first superficial observation ("grain is grain"), many different species and varieties, as well as differences caused by soil types, availability of local nutrients, make "grain" a significantly heterogeneous commodity, which requires special attention when sampled at various process locations (from harvesting, storage until commercial intake). The present appraisal shows that most of the respected HGCA grain guide's recommendations do not comply with TOS principles of sampling correctness. The suggested sampling procedures constitute major error potentials, which strongly compromise sample representativity.

## Introduction

The "Home Grown Cereals Authority" (HGCA) is a division of the "Agriculture and Horticulture Development Board" (AHDB) based in the UK, which is mainly responsible for research and knowledge transfer in the cereal and oilseed sector. As a private entity, the board of the AHDB and HGCA consists of grower and processor representatives, respectively, with an aim to "deliver a world-class arable industry through independence, innovation and investment".<sup>1</sup> In 2013 the HGCA published a guide on grain sampling to define key requirements for effective grain sampling at various process locations from harvest, to storage until departure and arrival of the grain.<sup>2</sup> Besides physical extraction of a grain "sample", focus is also on monitoring moisture, temperature, pests and moulds, especially mycotoxins. The delineated sampling practices must therefore ensure procedures that reliably are able to assess harvested grain quality, to protect this quality level throughout the storage phase as well as to determine quality level after storage (before transportation to buyer) and upon arrival at the buyer. For various commodities the latter two aspects (differences in quality level at departure vs quality level at arrival) have in the past caused major law cases, not seldom due to inappropriate or inadequate sampling procedures. Besides such discrepancies causing serious economic disputes, extraction of representative grain samples is also crucial with regard to impurity detection (e.g. GMO quantification, toxins), as regulated by international standards (e.g. ISO 24276:2006).<sup>3</sup>

The following critical assessment of HGCA's grain sampling guide serves to

evaluate whether *representative sampling* as delineated fully in the "Theory of Sampling" (TOS) is guaranteed when applying the guide's sampling procedures. Sample extraction, mass reduction and sample preparation are assessed for all process locations mentioned in HGCA with respect to the principles for representative sampling as set forward in TOS. All observed incorrect sampling errors are pointed out (incorrect *delineation*, —*extraction* and —*preparation*), which all raise the potential for an uncontrollable, inconstant sampling bias, jeopardising sample representativity. The present appraisal follows the principles laid down in a similar endeavour regarding a new standard for sampling of biomass.<sup>4</sup>

## Evaluation of suggested sampling procedures

Grain is a significantly heterogeneous commodity with a large amount of different varieties. The grain sampling guide points out that grain quality might be further affected by variation in "soil types, local nutrient availability [...], sowing dates, hedge and boundary effects and late tillering".<sup>2</sup> Besides such variation during the growing phase, especially the moisture content is affected when the grain is harvested and delivered to the storage facilities, depending on the weather and drying conditions. Additionally, mycotoxins might have affected parts of the grain load. Once stored in heaps, drying procedures can further increase variations in moisture level. The guide suggests to separate grain lots in "similar quality" units of 100t to decrease such variations, however, acknowledges that such strict separation of grain lots is not always possible due

to storage and on- and offloading procedures and conditions.

As a basis for the current appraisal Table 1 compares definitions of the basic sampling terms as used in the guide opposed with TOS' authoritative understanding of these terms, DS 3077.<sup>5</sup>

HGCA defines a representative sample, as a "final, well-mixed aggregate sample taken at one point in the grain chain". While there are some agreements with the much more elaborate definitions in TOS, the scope and focus is alarmingly narrow as shall be demonstrated.

Besides lack of several basic sampling terms, it is highly noteworthy that the term "accuracy" is wrongly defined in the HGCA guide (*sic*). Accuracy is a property of the mean, while precision is a property of the variance (TOS). Increasing the number of samples (increments), as stated in the HGCA guide, can only increase the precision (by decreasing imprecision), but has no automatic influence on accuracy. Accuracy can in point of fact only be ensured by following TOS' principles of sampling correctness, requiring that all bias-generating errors (termed "Incorrect Sampling Errors") be eliminated, DS 3077.<sup>5</sup> Furthermore, a correct (accurate) sampling process also needs to obey TOS's "Fundamental Sampling Principle" (FSP), which states that all units (particles, grains, fragments) in the lot must have an identical, non-zero probability of ending up in the final sample—implying that units not belonging to the lot must have a zero probability of being selected for the sample.<sup>5-8</sup> For practical sampling the above must also hold for the operational unit, the "increment". The FSP condition is missing entirely with HGCA.

**Table 1.** Basic sampling terms—Comparison HGCA vs TOS.

Sampling term*	HMCA grain sampling guide	Theory of Sampling (TOS)
Increment	“Incremental sample: any single sample taken by spear, jug or other means, to be combined with others”	Correctly delineated and materialised unit of the lot which, combined with other increments, provides a composite sample. For process sampling (1-D sampling) the only correct increment is a complete slice of the material, bounded by strictly parallel edges.
Composite sample	“Aggregate sample—a large sample comprising all smaller samples (i.e. incremental samples) taken at one point in the grain chain”	Correctly extracted material from the lot, which must only originate from a qualified “correct” sampling process being based on composite sampling
Representative sample	“A final quantity of grain from the aggregate sample using appropriate mixing/sampling procedures”	A sample can only be representative if the sampling selection process is <u>both</u> accurate (systematic part) and reproducible (random part)
Accuracy	“The more samples that are taken, the closer the average will be to accurately reflecting any characteristic”	A sampling process can only be rated as accurate if the average error $m_e$ equals zero, or a low value below an acceptable predetermined threshold: $ m_e  \leq  m_0 $ implying that for $ m_e  >  m_0 $ the sampling process is said to be <i>biased</i>
Precision	Not defined	A sampling process is said to be precise, or reproducible, if the variance of the sampling error is below a predetermined threshold level $\sigma_e^2 \leq \sigma_0^2$
Lot/ sampling target	Not defined	The complete entity of the original material being subject to sampling e.g. truck load, railroad car, process stream, ship’s cargo, batch. The lot (sampling target) refers both to the physical, geometrical form and size, as well as the material characteristics of the material being subject to sampling
Lot dimensionality	Not defined	TOS defines one-, two- and three-dimensional lots as well as the special case of a zero-dimensional lot, characterised by the effective number of dimensions involved in sampling

\* For all terms defined by TOS, see DS 3077<sup>5</sup> and references herein.

Also using a “jug” for increment extraction is a classical grab sampling procedure (see Figure 1 below), which far from always allows to cover all lot dimensions. Even in the ideal, optimal case of one-dimensional lots in which one dimension of the physical aspects of the lot dominates (e.g. material on conveyer belts, falling source streams), grab sampling is unacceptable; the situation is discussed thoroughly in DS 3077.<sup>5</sup>

Applying grab sampling to TOS 1-D lots in practice makes such lots 3-D, since singular grab samples are most likely taken from the surface part of the moving material flux, and almost certainly never covering both transverse lot dimensions entirely (contradiction to TOS’ Fundamental Sampling Principle), Figure 1. Any method involving manual shovelling, grabbing or similar simplistic material selection must be rated as *unacceptable*, since it unavoidably causes major Incorrect Sampling Errors.

### Primary sampling

In the following all sampling procedures of the HMCA’s grain sampling guide will be assessed and appraised according to whether they give rise to a high, medium or

low sampling error potential. Table 2 gives a summary of the evaluation results with respect to potential TOS-incorrect sampling errors.

### Sampling at harvest

The first sampling location in the grain transport pathway described in the guide is “sampling at harvest”, i.e. before the grain is

gathered in a storage/silo. The main aim of sampling at this process location is to give the buyer an early indication of the potential grain’s market value. Two different methods are outlined, one aiming at sampling grain before cleaning and drying, which takes place during the unloading of the trailer, the other sampling procedure aims at extracting samples from the cleaner/dryer outlet,



**Figure 1.** Examples of unacceptable manual grain grab sampling from 1-D moving lots. The left illustration suffers from severe accessibility issues, while the right illustration is overwhelmed by the material flux. Neither of these ‘incremental’ sampling procedures will make up to a representative aggregate sample.

**Table 2.** Potential incorrect sampling errors in HGCA's grain sampling guide.

Process location (HGCA)	IDE*	IEE**	IPE***
Sampling at harvest			
Method 1: Sampling before cleaning/drying — Sampling of trailer as it is tipped into store	High error potential	High error potential	Low error potential
Method 2: Sampling after conditioning — Sampling from the cleaner/dryer outlet	High error potential	High error potential	Low error potential
Sampling in store			
Sampling spear (3–5 apertures)	High error potential	Medium error potential	Low error potential
		Low error potential	
Sampling at outloading			
Sampling from loading bucket	High error potential	High error potential	Low error potential
Automatic bucket sampler	High error potential	High error potential	Low error potential
Sampling from spout loading Jug/Bucket Interrupter plate	High error potential	High error potential	Low error potential
	Medium error potential	Medium error potential	
Sampling from grain heap	High error potential	Medium error potential	Low error potential
	Medium error potential	Low error potential	
Sampling at commercial intakes Manual or automatic sampling spear	High error potential	Medium error potential	Low error potential
	Medium error potential	Low error potential	

\* IDE = Incorrect Delineation/Delimitation Error

\*\* IEE = Incorrect Extraction Error

\*\*\* IPE = Incorrect Preparation Error (refers only to primary sampling—mass reduction procedures are discussed further below)

i.e. after conditioning of the grain. As stated in Table 2 for both sampling methods the potential error for incorrect sample delineation and extraction is rated as high, while an incorrect preparation error is unlikely to occur. The IPE is, however, only rated for the primary sampling extraction at this stage, excluding further mass reduction steps, which will be assessed separately below.

Method 1 suggests extraction of two 500g samples from the trailer as it is tipped into the storage facility. “Ideally” these samples should be collected during the first quarter and the third quarter

during unloading (*de facto* acknowledging significant longitudinal heterogeneity in *any* trailer, and by implication in *any* 1-D lot). The sampling equipment, which should be used for this procedure is *not* defined, however. The guide only gives an overview of the sampling equipment needed for all sampling locations, stating sampling spear and measuring jug as the only equipment required for sample extraction. Since this particular sampling situation does not allow the use of a sampling spear (only applies to stationary lots), it must be assumed that using a measuring jug is the proposed sampling equipment for

method 1. Holding a measuring jug into the falling source stream does not allow *correct* sample delineation, dramatically disobeying the Fundamental Sampling Principle, Figure 1 (right). Even in case in which the jug is manually moved through the entire source stream, correct sample delineation cannot be ensured (compare DS 3077<sup>5</sup>). Furthermore, it is obvious that any hand-held jug will be filled very quickly due to the high mass flow during unloading. There will invariably be massive spilling-over effects, which only increase the stated high error potential for incorrect extraction. The storage of the primary sample is described correctly in the guide (low error potential for IPE), requiring a sealed plastic dustbin, which prevents loss and contamination of the sample.

Method 2 aims at extracting samples after the conditioning phase of the grain, for which “frequent” samples (around 250g every 10 tonnes) should be extracted from the outlet flow of the cleaner/dryer. As for method 1, the required sampling equipment is again *not* specified. Even though the second method implements a somewhat working process of incremental sampling (however, upward limited to only a total of 10 samples/increments), the unspecified sampling equipment is also here leaving the measuring jug as the only option. This again raises a high error potential for both sample delineation and extraction. Method 2 also mentions the option for using an “automatic bucket sampler”, in case the grain is moved into a bulk after conditioning. The automatic bucket sampler is evaluated below in the section for “sampling for outloading”.

### Sampling in/from storage

The second HMCA sampling location describes sample extraction from heaped, or piled grain lots in a storage facility; collecting samples from this location is only required in case samples have not been extracted during unloading of the trailers. The guide suggests use of a sampling spear with 3–5 apertures, but at the same time states that “such sampling is less likely to be representative of a given bulk than samples taken as the store is loaded” since sampling spears “cannot reach through deeper bulks/bins” due to their limited size range from 1.5m to 2m.<sup>2</sup> This inference by the guide is very much correct, see DS 3077 2013,<sup>5</sup> and is the reason the potential error for incorrect increment delimitation is rated as high in Table 2. Besides the very

limited accessibility of the grain located at the lower, and bottom parts of any pile or heap, the guide does not acknowledge that also a spear sampling requires incremental extraction, spread over the entire horizontal and vertical dimensions of the lot. The medium to low IEE error assessment is caused by the limited specifications of the sampling spear.

Even though the guide does not state explicitly for the “sampling in store” process location how to treat the extracted sample, it is assumed that the sample is to be stored in sealed dustbins as described for the first sampling location (low error potential for IPE).

### Sampling at outloading

The grain sampling guide recommends for this sampling location that it is best to extract samples from each lorry before departure—this is in full accordance with TOS. Assuming that a lorry load contains around 30t, the guide states to take at least 10 samples (each 200g), using one of the following sampling procedures.

As the optimal method for gaining a representative sample, the guide suggests to use a bucket or alternatively an “automatic bucket sampler”. Manual extraction using a bucket was described, assessed and denounced above, as a procedure, which can never lead to a representative sample and will always have a very high error potential (IDE and IEE). The “automatic” bucket sampler option described in the guide can be best understood as a classical bucket of a front loader, with the difference that “the bucket has another smaller opening (see Figure 2 below), which allows extraction of only a smaller portion of the material collected inside the bucket”. But the automatic bucket sampling procedure is also a grab sampling procedure, just in



Figure 2. ‘Automatic bucket sampler’  
(Source: HGCA 2013b).

larger scale, again risking a major error potential for IDE and IEE.

The third option described for the “outloading” sampling location is “sampling from spout loading”, referring to the loading position where the grain is transported on a conveyer belt into the lorry. Everyone familiar with the basics of the Theory of Sampling would immediately notice that this presents an optimal location for extracting representative samples, since the lot dimension due to the transportation on the conveyer belt, is reduced from three to one-dimensional. Once the grain falls from the conveyer into the lorry, the entire source stream can be correctly cut (sampled) using one of several types of *cross stream cutters*. This scenario is a classic example of sampling from a dynamic 1-D lot, extensively treated all over the TOS literature. HGCA’s grain guide, however, limits its recommendations to sample instead from a point close to the loading location, again *not* defining the used sampling equipment. In case a jug or bucket is used (grab sampling), a high error potential for IDE and IEE arise. Alternatively the guide mentions the use of an “interrupter” plate, which can be inserted into the conveying stream. However, neither the procedure nor the design of such interrupter plate is further described.

In case the interrupter plate is designed correctly according to TOS covering both width and depth of the conveyer belt, and the loading procedures allows to stop the conveyer belt at regular intervals, such “stop-belt” sampling procedure can be rated as satisfactory. However, due to the lack of specifications in the guide, the assessment in Table 2 rates the interrupter plate option with a medium error potential for IDE and IEE.

The last sampling procedure suggested in the guide during outloading describes sampling from a pre-positioned grain heap, which will be subsequently filled into a lorry. A sampling spear is again suggested for sample extraction in this situation. Similar to the critique raised under “sampling in store”, the error potential in particular for correct delineation depends on the height/size of the lot versus the length of the sampling spear. In case the applied sampling spear does not reach to the full depth of the grain heap, sampling correctness is of course also here strongly compromised (high error potential for IDE) and therefore unacceptable.

### Sampling at commercial intakes

The final sampling location described in the guide aims at sampling at commercial intakes, required to check whether grain quality meets the agreed contractual requirements and specifications. For this sampling location the guide refers to the ISO 24333:2009 standard for sampling cereals and cereal products, which again recommends a sampling spear to extract samples from the incoming grain across the lorry load. The standard correctly explains that the sampling spear must be “long enough to sample the whole depth of grain”,<sup>9</sup> required to fulfil TOS Fundamental Sampling Principle. The FSP is still compromised, however, by subsequently stating that: “... the lorry should be positioned so that most of the load is accessible...”. Needless to say this lax “most of the load” requirement is an open invitation that causes biased samples.

The number of increments is generally fixed to eight samples per lorry, but only three for lorries of 15 tonnes or less. Since insertion of the sample spear, as well as total number of extracted increments, is strongly interacting with the empirical lot heterogeneity, the potential for IDE is rated as medium. The HGCA guide correctly states: “grain may not be uniformly mixed” and: “heaping in the vehicle [...] does not always level out during haulage and this can bias sampling”. In fact, it should be noted that road or rail transportation will cause materials to segregate significantly, leading to increased distributional heterogeneity (the exact opposite of “uniformly mixed”), which makes sampling position and total amounts of increments even more important. The rated error potential for incorrect sample extraction (IEE) is depending on the detailed design of the sampling spear involved.

Alternatively to a manual sampling spear the grain guide suggests the use of an “automatic sampler” (automatic sampling spear), for which the same evaluation results apply as for the manual sampling spear if used in the same fashion under identical adverse conditions (see Table 2).

However, there exists a very good alternative “automatic spear” sampler, in the form of what is known as the “RAKORAF” sampler.

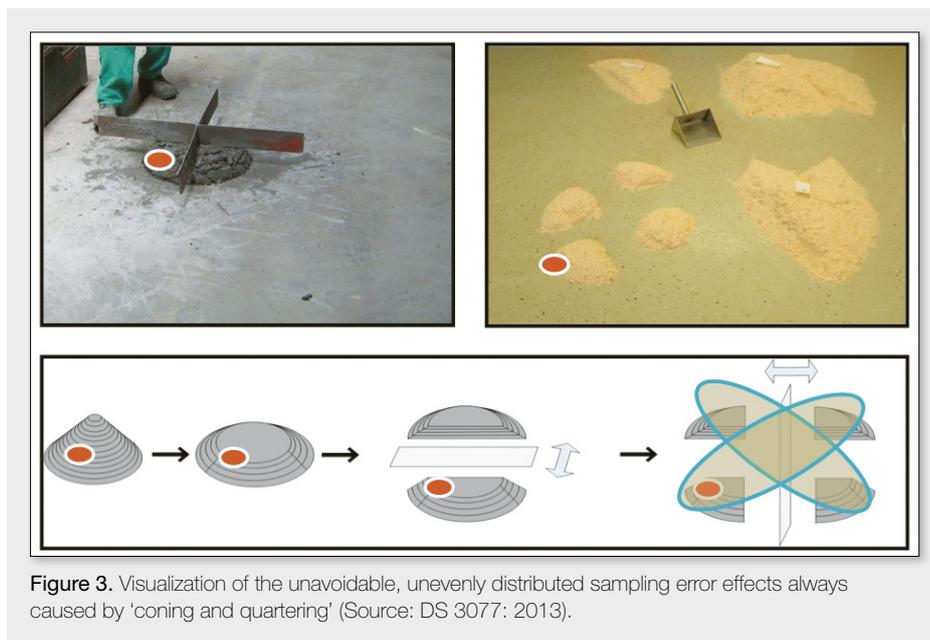
The “Rakoraf Core Sampler” (RAKORAF) allows automatically to extract representative increments or samples from open grain truck trailers. A telescopic arm with a core tube is lowered into the grain

load, but not by forceful insertion. The gentle downward movement of the sampling tube allows grain kernels to enter into the inner chamber of the core tube, which subsequently transports the increment upwards into a topside receiving chamber, where air is separated from the extracted sample. The main difference towards the forcefully inserted automatic sampling spear is the fact that the RAKORAF has a zero-pressure differential across its opening aperture, which specifically avoids a so-called “vacuum cleaner effect”. Indeed the ingenious design feature allows perfect *isokinetic extraction* of a delineated vertical, cylindrical increment (see also Reference 4). Further information about the different versions of the RAKORAF can be found on the OEM’s website.<sup>10</sup>

### Mixing and subsampling

The Theory of Sampling provides the theoretical background as well as practical sampling approaches (termed “sampling unit operations”—SUOs) to acquire representative primary samples, as well as to guarantee sample representativity throughout all sub-sampling and mass reduction operations making up the full pathway from lot to analytical aliquot.<sup>11</sup> As correctly stated in the grain guide “it is important to ensure, as far as possible (*sic*), that all grains in the aggregate have an equal chance of being included in any sub-sample drawn from it”.<sup>2</sup> The equal likelihood for units to be selected is of course not an option (“as far as possible”), but an imperative requirement for ensuring representativeness for both the primary sample extraction stage and in all stages until the final aliquot mass has been extracted. The HGCA is too lax in its requirements.

To acquire a valid sub-sample size, the grain guide first states to “thoroughly mix” the aggregate sample (composite sample) by using a drum mixer (sample is placed in a drum and rolled around its axis) or by spreading the sample on the floor and manually mixing it using a shovel/scoop. Many studies have shown, however, that mixing often only has limited effects on the distributional lot heterogeneity. In general forceful mixing is far from the globally effective process often assumed, indeed may sometimes even causes an increase of segregation. Although very often diminishing heterogeneity, simply stipulating “mixing” is unfortunately not a universal guarantee for success in the next sub-sampling stage.



**Figure 3.** Visualization of the unavoidable, unevenly distributed sampling error effects always caused by ‘coning and quartering’ (Source: DS 3077: 2013).

After the mixing process the guide suggests, with reference to ISO 24333, to use “coning and quartering”, or to use sample dividers like cone-shaped divider, rotary mechanical divider or riffle divider, for reducing the sample mass. A very detailed comparative survey of various mass reduction techniques by Petersen *et al.* has shown that there are many pitfalls in the laboratory stage mass reduction game.<sup>12</sup> This comprehensive survey concluded that rotary dividers and riffle splitters are the only acceptable mass reduction techniques. The grain guide, however, *focuses* on “coning and quartering” and gives a detailed instruction on how to perform this non-acceptable mass reduction technique. We need here to take a very firm stand against any coning and quartering, at any scale.

In Figure 3 an attempt has been made to illustrate the general problem caused by coning and quartering. The two upper photographs show industrial use of a splitting cross (left picture) and a conventional shovel (right picture) to perform the quartering of the previously coned lot. The delineated (oval) designation in both pictures represents for example a high concentration of analyte (“hot spot”), which might have been caused by prior segregation effects or other. The lower figure shows that the designated volume may end up fully in one of the quarters (or it may be unevenly divided in two neighbouring quarters). No matter which of the two opposed quarters is chosen to make up a 50/50 subsample, the analyte

concentration of the lot is either over- or underrepresented, always causing a biased subsample (except in the ideal 50/50 hot spot split case, which is so far from the general case as to be any interest).

### Conclusions

Assessment of HGCA’s grain sampling guide shows that most of its recommended sampling procedures, and equipment (for both primary sampling and sub-sampling) do not lead to a representative sample. The guide’s sampling procedures have a high error potential for incorrect sample delineation and extraction, which unavoidably will lead to a significantly detrimental, or even fatal sampling bias.<sup>4</sup> While for all stated grab- and shovelling methods, sample representativity can hardly ever be ensured, the remaining sampling procedures, some of which may be somewhat constructive, were it not for the fact that they very seriously lack sufficient specification, inevitably increases the potential for incorrect sampling error effects. Most of the guide’s recommended sampling equipment, when rated with TOS criteria, reveal major incorrect sampling errors (ISE), vastly jeopardising grain control validity. The only exception from this conclusion concerns riffle—and rotary splitters also recommended for sub-sampling (but to its chagrin, HMCA stresses coning and quartering).

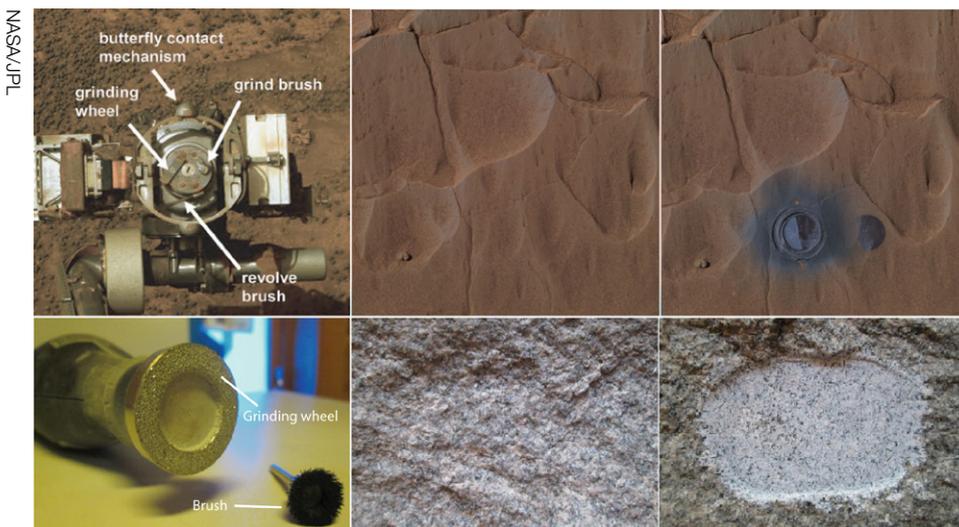
Only *representative samples* can serve for quality control, preventing disputes between grain producer, seller and buyer. There is no declination of the adjective

“representative”—either a sampling process can be documented to be both accurate and sufficiently precise, representative, or it cannot.<sup>4</sup> It is strongly recommended to integrate TOS’ basic concepts for sampling representativity in HGCA’s grain sampling guide, without which efforts towards representativity are in vain. A comprehensive and complete TOS-approach to grain sampling from “large kernel lots”, was published recently,<sup>13–15</sup> which along with the selected TOS literature referred to above, gives a complete roadmap how this can be accomplished.

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## Coming up in future issues



### Sampling on Mars inspires development back home on Earth

Inspired by the RAT (Rotary Abrasion Tool) on board the Mars exploration rover Curiosity (upper panel illustrations), a recent engineering thesis by Munim Morshed (2014) Telemark University College, Porsgrunn, Norway (2014), examines the possibilities of producing a FRAT (Field Rotary Abrasion Tool) (lower panel) intended to prepare rock surfaces for improved handheld XRF and NIR analysis in the field back home on Earth.

A summary of this thesis will appear in one of the next issues of *TOS forum*.



### Sampling on Mars—exactly like on Earth?

A recent thesis by Munim Morshed, Telemark University College, Porsgrunn, Norway (2014), examines the fleet of Mars exploration lander/rover sampling equipments, from a strict Theory of Sampling (TOS) perspective. There is a clear evolution from grab sampling (Viking landers) to fully correct sampling. Pictured here is an example of correctly sampled drill cores made by the contemporary rover Curiosity. A summary of his findings will appear in *TOS forum*.

# Visualisation of sampling error effects in near infrared analysis—comparison between Petri dish, roll bottle and spiral sampler

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With spectroscopic methods, e.g. near infrared (NIR) analysis, using a constant beam aperture, the effective scanning footprint will be different for a spinning Petri dish, a rolling bottle and a new spiral sampler configuration. This will significantly influence the analytical accuracy and precision of a NIR analytical determination of heterogeneous materials, for example barley with differing protein contents. Here we present the results from a bench-top experiment that evaluates the total analytical bias and precision characteristics for three alternative sample presentation approaches using a mixture of two plastic polymer pellets as a test material with significant heterogeneity. After removal of all incorrect sampling errors (ICS), there are still significantly varying correct sampling error [Fundamental Sampling Error (*FSE*) and Grouping and Segregation Error (*GSE*)] uncertainties associated with these standard analytical approaches—but there is a clear winner.

## Introduction

Analytical spectroscopic scanning methods such as near infrared (NIR) spectroscopy are widely used to analyse a multitude of different types of materials, most of which are irregular (heterogeneous). Based on proper calibration and validation with respect to a suitable reference method, NIR analysis is a fast approach giving reliable results in a very short time (often <1 min), hence its enormous range of applications and number of successes. Much interest is traditionally given to the analytical performance of the instrument and this may vary depending on the instrument type, the acquisition conditions and the quality of the calibration. In many applications, however, this is of less importance if analysis is to be performed on materials with a significant degree of *heterogeneity*. In such a situation, it is of essential interest to focus both on how the analytical aliquot was produced (representativity of the full sampling process) as well as how it is introduced to the instrument. Most common laboratory instruments either use fixed volume vials or Petri-dishes for liquid and solid samples, respectively. While these are adequate methods for sample materials which are compositionally relatively uniform (e.g. one-phase liquid samples, finely ground, well-mixed materials and/or natural powders), significantly heterogeneous material like whole wheat, grass, hay, silage, meat, vegetables, fruits, berries etc. will present a severe challenge due either to particle size and/or degree of heterogeneity

in relation to the absolute sample size possible (often fixed by the Petri dish diameter). A representative sample will often be difficult to define in such cases if not based on the full principles of the Theory of Sampling (TOS). In many analytical communities there has been, perhaps understandably, a tendency to the attitude of “too much of this sampling focus—let’s get on with the analysis”. It would, for example, be quite pleasant were such issues to be eliminated by a “smart, fit-for-purpose” sample presentation technique or if a universal accessory was available. At various times the spinning Petri dish, as well as the rolling bottle and other adaptations have both been hailed as more or less the final answer to these issues, and the newly

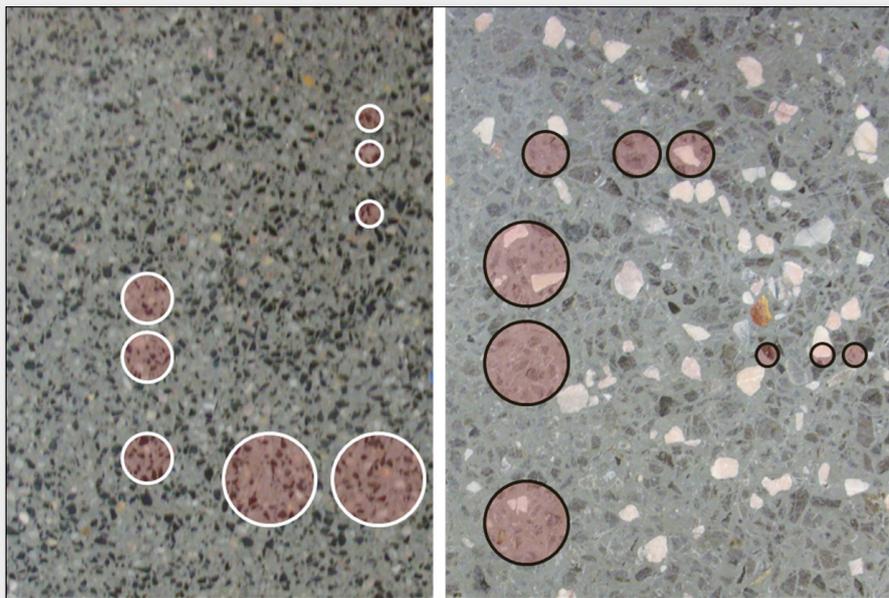
developed “spiral sampler” is but the latest such candidate.

It was felt that it would be useful to both the NIR and sampling communities to compare these three widely-available sample presentation options based on a quantitative evaluation. We shall analyse the sample presentation and spectral acquisition situations from the principles of the TOS, but otherwise let the numbers speak...

Three alternative sample presentation approaches compared in the present study are shown in Figure 1. The effective sample mass (area) achieved in each is markedly different. The predominant effect and difference between the accessories are the observable surface, and hence sample



**Figure 1.** Illustration of the different NIR accessories [Petri sampler (left), bottle sampler (centre) and spiral sampler (right)] which present the sample to the spectrometer in three different ways. The effective scanned areas are also indicated (dependent on method/number of scans) and the maximum sample volume for the respective sample containers.



**Figure 2.** Illustration of the effect of increment size (circles) for two materials with markedly different heterogeneity. In NIR analysis the increment size can be increased by acquiring a larger sample area (a larger circle area in this illustration). Using “too small” a sample area (small circles) can clearly lead to significantly different analytical results when performing repeated analysis, due to significant *GSE* and *FSE*.

sampling will never result in exactly identical concentration values. Repeated sampling, even with exactly the same procedure, will unavoidably extract *different* increments of the heterogeneous lot material. This is the effect of what is termed the Fundamental Sampling Error (*FSE*) in TOS. In practice, the effect is often augmented by uncertainty stemming from the Grouping and Segregation Error (*GSE*) which originates from meso-scale spatial heterogeneity. The primary way to reduce *FSE* and *GSE* is by reducing the degree of heterogeneity, which can be done effectively by comminution and subsequent mixing or just mixing (to reduce the existing spatial heterogeneity). Alternatively a larger sample size has to be used—or best, a combination of all of the above.

What is needed in a specific situation will be determined by the decision of the acceptable level of the *TSE* (Total Sampling Error)—to which must always be added the Total Analytical Error (*TAE*). The main issue is that *TSE* is nearly always significantly larger than *TAE* (factors of 5–20 are normal and may be even higher in particularly adverse situations). *FSE* can be estimated mathematically following Pierre Gy’s formula<sup>1–3</sup> which, with *some* approximation, can be used to estimate the minimum sample mass, or conversely, the maximum

grain size that corresponds to an *a priori* given (*TSE* + *TAE*) level. In general, *FSE* is inversely correlated to the effective aliquot mass being analysed and is also dependent upon the analyte concentration. However, the influence of *GSE* is **not** included in any of this type of *FSE* estimation, for which

reason many practitioners prefer to establish the *effective* sample mass vs acceptable *TSE* + *TAE* levels by some appropriate *empirical* approach; this forms the basis for the present evaluation. For a more in-depth introduction to the specific TOS issues, see, for example DS 3077<sup>4</sup> and the many references found therein.

### Materials and methods

White polyethylene (PE) pellets and white polystyrene (PS) pellets were purchased from industrial plastic manufacturers. The density of each type of pellet was determined experimentally. Master samples of 2%, 10% and 20% PS concentration levels in PE (vol/vol) were prepared based on appropriate masses and pellet densities. The accuracy (absolute level) of these concentration levels 2%, 10% and 20% is not important with respect to the conclusions but all master samples were still prepared with the outmost care, allowing us to assume the error contribution from preparation can be neglected in this experiment when compared to the errors arising from sampling/presentation. Sampling from master sample lots was carried out using a spoon with a size chosen such that the volume in each type of sample container was achieved by combining 10–12 composite increments. The filling degree was 100% for all containers in the present study.



**Figure 3.** Illustration of the different sample masses typically achieved with the Petri dish (left), rolling bottle (centre) and spiral sampler (right). Typical proportions between the effective analytical masses are 35 g, 50 g and 600 g, respectively. N.B. the analytical NIR sensor system does not interact with the entire mass in the containers.

NIR scanning was performed on a Quant FT-NIR instrument (Q-Interline, Tølløse, Denmark), equipped with an InAs detector. The spectra were acquired in the range from  $4000\text{ cm}^{-1}$  to  $12,500\text{ cm}^{-1}$  ( $800\text{--}2500\text{ nm}$ ) with a data point spacing of  $8\text{ cm}^{-1}$ ; 180 scans were recorded for each analysis. For the needs of the present comparison experiment, all analysis was carried out under the same ambient conditions.

## Experimental design

The experimental design (Figure 4) imitates sampling from a realistic primary lot followed by packing in container and analytical measurement. Each of 10 sampling replicates from the master lot was measured with the standard NIR approach (experimental level S1). One random replicate was re-packed and measured 10 times (experimental level S2). Finally, one randomly selected replicate was measured 10 times to estimate repeatability (experimental level S3). This design was applied identically to all three sample presentation methods.

## Spectral data prediction model

To predict all concentrations in this binary experiment, a PLS1 calibration model was constructed. Samples used for calibration comprised a single spectrum from each sample presentation method and each concentration level in this study, plus spectra of four additional concentration levels. The spectral range used was  $5870\text{--}9025\text{ cm}^{-1}$

and pre-processing comprised Savitzky–Golay 1<sup>st</sup> derivate, 13 points. As could be expected, the model needed only two PLS components to explain close to 100% of all variation. This model was used to predict all the PS concentrations in the study.

## Results and discussion

For an apparently simple and straight-forward analytical method, such as NIR, there are still multiple error sources that contribute to total uncertainty variance. The primary sampling error effects are associated with how to get a representative sample with respect to the whole lot without contributions from incorrect sampling errors. Here, the total sample mass must be considered relative to the inherent heterogeneity of the lot. Very heterogeneous materials would of course benefit from a larger sample size as compared to more uniform materials (but **only** if based on proper composite sampling). This is needed in order to get an acceptable, reduced contribution from *FSE* and *GSE*. If sub-sampling must be employed, each such stage forms a completely new “primary sampling” scenario at a reduced scale. Petersen *et al.*<sup>3</sup> present a complete survey of all available techniques for this purpose, including empirical evidence for selecting optimal approaches only (splitting).

At some point, the proper sample size (mass) has been achieved, however, and is now to be presented to the NIR instrument. This also has to be carried out in a

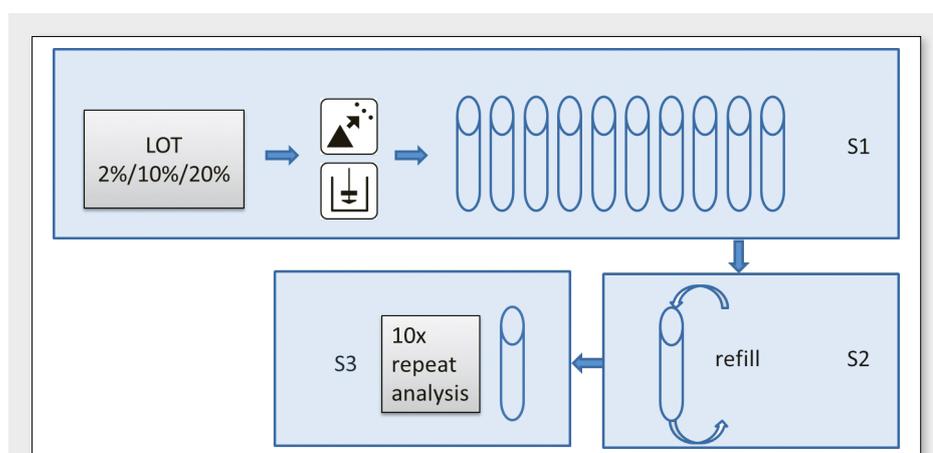
representative manner, i.e. all parts of the scanned aliquot (sample) should have the same probability of contributing to the analytical spectrum. This is often not the case with the three options being investigated. This is often a direct effect of the type of sample preparation, forced by the design of the sample presentation method and accompanying sample container (Petri dish, bottle, spiral sampler tube). Additionally, the spatial filling of these sample containers can contribute to *GSE* as there may be a tendency towards different packing as a result of differences in density, surface properties and shape of particles in the lot material.

The final focus for the present study is the sample presentation methodology, which will influence the validity of the analytical results with regard to effective scanning area relative to sample size (a *FSE* issue) and the physical sample presentation that should seek to minimise effects from *GSE*. The *effective* scanning area is the area of the sample surface that is actually scanned in depth and which contributes to the acquired NIR spectrum. Typical proportions between the effective scanned areas are  $18\text{ cm}^2$ ,  $30\text{ cm}^2$  and  $300\text{ cm}^2$  for the Petri dish, the bottle spinner and the spiral sampler, respectively.

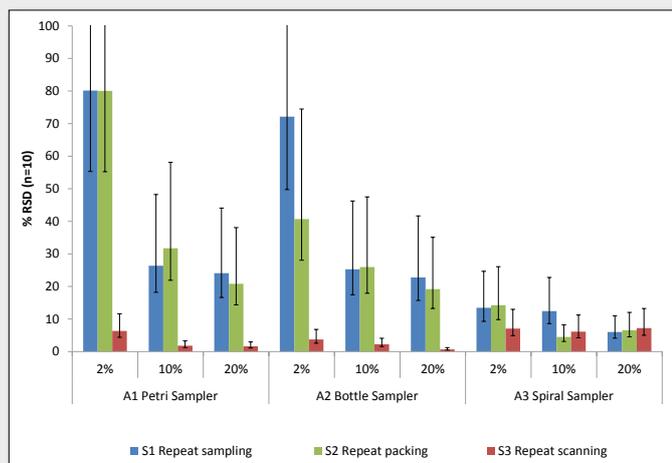
## Petri sampler

For the Petri sampler, the effective scanning area is an annular area measured on the bottom side of the Petri dish which in the present case corresponds to  $\sim 18\text{ cm}^2$ . Increasing the number of scans above the acquisition time corresponding to a full annular revolution will not reveal any new sample surface area but merely results in repeated scanning of the *same* sample surface as has already been fully covered. As a result, multiple measurements (S3) of the same Petri dish give excellent repeatability (Figure 5). However, if the sample is re-packed or re-sampled in the Petri dish, a completely different result is revealed. This can be seen in Figure 5 as a significantly larger standard deviation for re-packed and re-sampled Petri dishes (S1 + S2) compared to repeated measurements (S3).

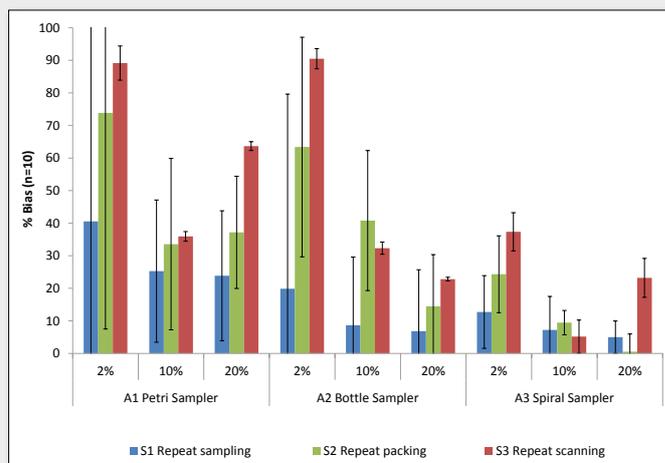
Relatively large bias values are also characteristic for the Petri dish presentation (Figure 6); this does appear also to be the result of the small scanning area. Combining the reproducibility (here repeatability) and bias into representativity, a central



**Figure 4.** Flow-path diagram of the experimental design. Experimental level S1: the three master sample lots at 2%, 10% and 20% PE were mixed thoroughly and then, using composite sampling, 10 sample containers (Petri dish/rolling bottle/spiral spinner tube) were filled and analysed by NIR with each of the respective sample presentation methods. Experimental level S2: after NIR analysis, one of these sample containers was emptied and re-packed into the same container 10 times, each analysed by NIR. Experimental level S3: one container was finally re-analysed 10 times. Each cylinder in this illustration represents either a Petri dish, rolling bottle or spiral sampler tube.



**Figure 5.** Replication error contributions shown expressed as relative standard deviations obtained for 10× repeated sampling, 10× repeated packing of sample containers and 10× repeated measurements for each sample presentation method and at each of the three concentration levels. Error bars show 95% confidence limits.



**Figure 6.** Bias contribution shown as relative bias [%] as obtained for 10× repeated sampling, 10× repeated packing of sample containers and 10× repeated measurements for each sample presentation method and at each of the three concentration levels. Error bars show 95% confidence limits.

tenet of TOS, one reaches the conclusion that the Petri spinner has the worst representation of “the truth” which is known in this controlled experiment (Figure 7). The representativity quickly becomes devastatingly worse at low concentrations because of the progressive influence from the irregular distribution of the analyte (increasing GSE). This feature disqualifies the Petri dish as a valid element in any *representative* measurement system.

This is exactly what should be expected in the light of TOS. Since the Petri dish is measured on the base, what is measured is dominated by material that settles at the bottom. For sample material prone to segregation (very many types of foods, feeds, powders) this results in significant GSE and may cause a large bias as well. Because of this risk of segregation and the limited size of the dish, the Petri sampler is only suitable for fine powders and even here thorough mixing is a requirement; for more heterogeneous material, the contribution of GSE will reduce analytical performance rapidly.

### Bottle sampler

The bottle sampler accessory is designed to rotate a semi-filled 125 mL bottle around its length axis while positioned at an angle of 22° to the horizontal; this offset is in order to stimulate tumbling/mixing (however, in this study the bottle spinner is used with 100% filling for comparative reasons). The scanned area is the circular belt area

around the cylindrical sides of the bottle, which corresponds to a mere 15 cm<sup>2</sup>. But, for bottle contents that mix during spinning, increasing the number of scans will, to *some degree*, increase the effective scanning area as long as scanning continues. For a full bottle, however, the effective total scanning area will not increase above 15 cm<sup>2</sup> and in this regard the bottle sampler should then resemble the Petri dish and therefore show similar trends for standard deviation and bias (Figures 5 and 6) which is indeed the case. Compared to a Petri sampler, there is a tendency for a lower bias (not significant) for the bottle sampler. Since the glass quality is similar and all other parameters are kept identical, this difference could relate to the target material. The plastic pellets are perhaps packing better in the bottle as compared to the Petri dish bottom. The end result (Figure 7) is that bottle sampler representativity is slightly better than the Petri dish, especially at higher concentrations which is in support of the thesis that the pellets are not perfectly identical and hence pack differently. These minor differences will of course be larger with increasing contrast between different particle sizes and/or densities.

Detailed inspection of Figures 5 and 6 reveals minor differences in the relationships between the performance of the rolling bottle and Petri dish with respect to both bias and replication variability. These differences are not statistically significant

but are stochastic reflections of the interplay between heterogeneous materials being repeatedly sub-sampled, re-packed and re-analysed. No general conclusions can be drawn on this basis; there is always such a random effect in the sampling plus analysis system.

### Spiral sampler

For the spiral sampler, the scanned area is a belt wrapped around the glass tube in a helical fashion. This helical belt ensures that the beam footprint continues to cover new sample material along the entire cylinder length and effective scanned area is therefore limited only by measurement time and tube length. For the size of glass tubes used, the maximum possible area is 375 cm<sup>2</sup>. Experiments reported here were actually limited by measurement time (here 180 scans) corresponding to 275 cm<sup>2</sup>, which is still many times larger than for the other methods (>five times). Due to the larger scan area, the repeatability of scanning is not as low as for the other sample presentation methods (Figure 5, S3). While for both the Petri spinner and bottle sampler there were significantly higher standard deviations for repeated sampling (S1) and re-packing (S2) compared to repeat scanning (3), this is not the case for the spiral sampler. This means that one properly taken sample fully represents the heterogeneous lot under study and that repeated, repacked sampling reveals no new information. This

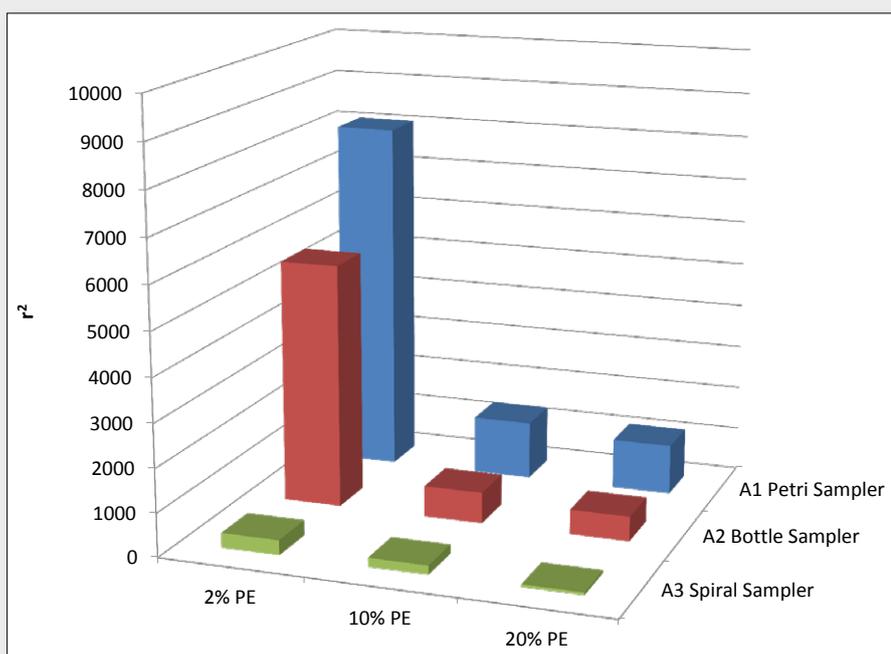


Figure 7. Total representativity characterisation for 10-fold repeated sampling, sample presentation and analysis using A1: Petri sampler, A2: bottle sampler and A3: spiral sampler.

becomes very clear when representativity calculations are done for the spiral (Figure 7) in which case the results are magnitudes better than for the other two methods.

Both the relatively large sample size and the large scanned surface area influence the *FSE* and *GSE* in a positive direction.

In this study, the spiral sampler was tested completely filled but can also be used with a fill level that enables mixing, as can the bottle sampler. Such a strategy will, in general, not change the present findings significantly.

## Conclusion

The three alternative sample preparation methods have very different characteristics in terms of precision and accuracy. It is evident, not only from the present results, that analytical precision alone is **not** an adequate measure of the performance of a NIR method as has otherwise been considered good practice for a while. Only after careful evaluation of the potential offset of the results, stemming from **both** *GSE* and *FSE*, may a particular NIR measurement system be successfully applied without significant risks of faulty and potentially expensive, wrong conclusions.

No doubt the well-known and easy-to-use Petri dish is the winner in the battle

for best precision under repeatability conditions, but the characterisation as best precision *per se* is a complete *mirage*. It is abundantly clear that this only reflects the ability to predict the same wrong result several times in a row (it is in fact simply analysing “precisely wrong”). The bottle spinner is, in general, slightly better although not hitting any highs compared to the new spiral sampler, which combines good precision with low bias and thus very clearly comes out on top with respect to a full definition of representativity. Its ability to lower *FSE* and *GSE* significantly is due to the much larger composite sample mass and a much larger effective scan area.

What happens to a sample received in the analytical laboratory is not a trivial matter; significant sample preparation and presentation errors can arise. Still, much will depend on the validity of the full sampling plus analysis process—i.e. the first sampling stage is of critical importance concerning the accuracy with respect to the original lot (potentially creating a significant, inconstant bias). The entire “lot-to-aliquot” pathway is analysed rigorously from the standpoint of TOS in a new international sampling standard, DS 3077.<sup>4</sup> The task of being able to produce correct predictions, i.e. accurate and precise predictions closely resembling the real world,

may often necessitate that the full chain of actions from primary sampling to NIR acquisition must be rewritten, away from what is the most “convenient” to what is most accurate and follows closely the principles of TOS.

The experimental binary “product” used here mimics many types of real-world counterparts and materials, for example freshly harvested sugarcane, silage, corn or the likes with stems, seeds and fragments displaying areas close to, or even larger than, one singular NIR beam footprint. For all such material types, as well as for all materials with *similar* heterogeneity characteristics, the clear winner is the spiral sampler.

Further, there is a significant potential for transfer of the present results to other applications within the area of process quality control employing PAT, often also using fixed-beam NIR sensor technologies, whether in-line, on-line or at-line. The same TOS principles invoked here can also be applied there, see, for example, Esbensen and Mortensen.<sup>5</sup>

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# Sampling 2014 Where it all begins



## Your Invitation



On behalf of the organising committee, I invite you to register for Sampling 2014 being held in Perth on 29–30 July 2014. It is the fourth in the conference series organised to bring together everyone involved in mineral sampling, including exploration, resource evaluation and mine development through to grade control, process control and commercial transactions. Once again, the conference is

being jointly organised by The AusIMM (The Australasian Institute of Mining and Metallurgy) and CSIRO.

The theme of Sampling 2014 is “Where it all begins”. Despite the use of quite advanced technologies in the minerals industry, it is still surprising how little attention is given to extracting samples for resource development, process control, plant optimisation and product sales. 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 of the ore or product, the whole analysis process is flawed at the outset. Unfortunately, no amount of re-analysis can fix this problem. As a direct result, mining companies stand to lose millions of dollars in terms of poor investment decisions, wasted mineral resources, poor plant recoveries and income from the sale of their products.

Join with us to share information with like minded professionals. The conference will provide valuable opportunities for networking, meeting respected international sampling experts, sharing ideas and catching up on the latest developments in sampling and sample preparation.

I look forward to seeing you at Sampling 2014 and hope that you take the time to register, engage and participate with other like minded specialists.

Sincerely,

**Dr Ralph Holmes**

Conference Chair, Sampling 2014

CSIRO Minerals Down Under National Research Flagship

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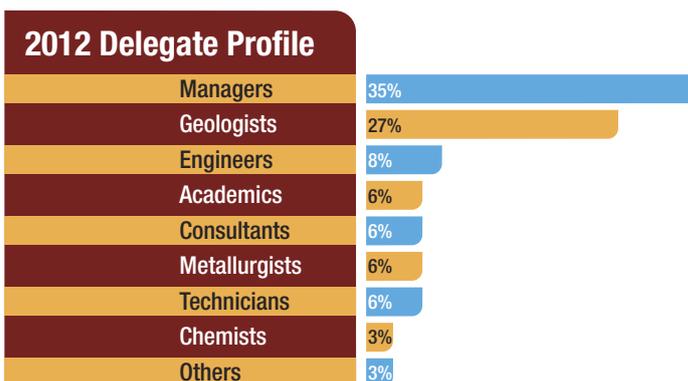
## WHY ATTEND SAMPLING 2014

- Connect with other professionals in the sampling community
- The chance to learn what others in the field are doing
- Address key issues, current and future trends
- Collaborate with industry experts
- Hear from inspiring keynote speakers
- Interact at a trade exhibition with prominent organisations
- Challenge yourself and attend additional thought provoking workshops
- Presenting a peer reviewed technical program experts, designed to highlight the specific issues which matter to you

## CONFERENCE THEMES

- Development and application of sampling theory
- Drill and blasthole sampling
- Plant sampling
- Sampling for blending, quality control and metallurgical accounting
- Sampling of commodity exports
- New developments in sampling and sample preparation equipment
- Maintenance of sampling equipment and training
- Development of national and international standards
- Case studies of the application of sampling in exploration, mining, mineral processing, export and environmental monitoring

## SAMPLING 2012 DELEGATE PROFILES





## Preliminary Timetable

Monday 28 July	Tuesday 29 July	Wednesday 30 July	Thursday 31 July
<b>W1: Concepts in the Sampling of Gold Deposits</b> 8.30 am – 5.00 pm  <b>W2: Sampling Basics Workshop</b> 8.30 am – 5.00 pm	<b>Sampling 2014</b> Conference and Exhibition		<b>W3: A Practical Guide to Designing and Running Effective Sampling Programs</b> 8.30 am – 5.00 pm
	<b>Networking Hour</b> 5.00 pm – 6.00 pm  <b>Conference Dinner</b> 7.00 pm for 7.30 pm		

## Preliminary List of Papers

### Case Studies of the Application of Sampling in Exploration, Mining, Mineral Processing, Export and Environmental Monitoring

Empirical Analysis of Sampling Methodologies for Gold in Soil and Stream Sediments — *D Arne*

The Influence of the Fragmentation Mechanism in Determining the Heterogeneity of Gold Ores — *A C Chieragati, H Delboni Jr, R Senefonte, H G Ernesto and T O Luna*

Metallurgical Accounting Issues with Toll Milling of Gold Ores — *D Connelly*

Time versus Mass Basis Sampling — *M Handyside and J Ta'ala*

The Application of Automated Samplers in Copper/Gold/Nickel Processing — *J Powalisz*

A Comparative Study of Manual Wagon Top Sampling and Auto Mechanical Sampler of Coal 200 mm Size with Respect to Stopped Belt of Thermal Coal at Indian Super Thermal Power Plant — *K M K Sinha, K K Sharma, G S Jha, K M P Singh and T Gouricharan*

When Nothing is What is Needed. The Importance of Using Blank Samples at all Stages of Sample Preparation — *J Webb*

Large Diameter Coring — *P Jacobs*

### Development and Application of Sampling Theory

Bulk Solids Sampling — *J Powalisz*

### Development of National and International Standards

Comparison of Two Kinds of Test Methods of Testing for Bias of Mechanised Sampling Equipment of Coal — *Z Wu and P Zhongyuan*

### Drill and Blasthole Sampling

Sources of Sampling Error and Implications for Quality Assurance and Quality Control in Surface and Underground Reverse Circulation Drilling and Cone Splitters — *J Carswell*

### New Developments in Sampling and Sample Preparation Equipment

Comparison of the Methods and Results of the Determination of the Fundamental Sampling Error (FSE) by Different Methods — *R Minnitt*

Control and Monitoring of ISO Compliance for Industrial Sampling Systems — *T Neidel, C Adams and R Shaw*

The Economic Benefit of RC-Assist Grade Control in Mining — *T Sheldon and T Day*

### Plant Sampling

Sampling Protocol Development in a Coarse Gold Deposit — *S Dominy*

Sampling Challenges In Modern Mineral Processing Plants — *C Evans and D Drinkwater*

An Investigation of Current Flotation Survey Sampling — *D Laskovski*

Analysis of Slurry Sampling Arrangements at a Froth Flotation Plant — *J Lohilahti, T Korpela and P Minkkinen*

Justification for Integrating Mechanical Sampling with Online Analysis — *D Stevens*

### Sampling for Blending, Quality Control and Metallurgical Accounting

Guidelines for Rational Subgrouping in Estimating Standard Deviation for Statistical Process Control — *W Breytenbach*

Plantwide Bias Detection Using Statistical Data Reconciliation — *L Lachance, S Gariépy, D Leroux and F Flament*

True Pipe Sampler – A Correct Design Utilising the Principle of Symmetry in Pressurised Slurry Particulate Streams — *R C Steinhaus*

### Sampling of Commodity Exports

Cape Lambert Port B (CLB) Ship-loading Sampling and Analysis — *R Brunning, C Andringa-Bate, M Graham and S Westergren*



## Keynote Speakers



**Steve Campbell**, President JBLCo, Inc. USA

### **Sampling Market and Trends in North America – The Shrinking Coal Market in the US: Sample More or Sample Less**

Steve is the president and CEO of John B Long Co (now JBLCo, Inc) of Knoxville TN. JBLCo is a small but diverse worldwide company with three operating divisions. JBLCo provides mechanical sampling systems for all types of bulk materials. With its start in the coal industry JBLCo has become a worldwide provider of equipment to sample products of all types.



**Dominique François-Bongarçon**, Proprietor, Agoratek International, Canada

### **State of Affairs and Current Research Trends in Theory of Sampling Fundamentals, Including Segregation and Correctness**

Dominique graduated as a mining engineer in France in 1974. He then obtained a Doctorate in Mining Sciences and Techniques, at the Paris School of Mines, in 1978. Since then, he spent several years as a Research Engineer at the Geostatistics Center in Fontainebleau, and after 1980 he worked for a number of international consulting firms and mining companies, including INCO Ltd in Canada. From 1992 until now, he then devoted his research time to Gy's Theory of Sampling.

In 2009, he was the recipient of the Pierre Gy Sampling Gold Medal. His two consulting companies, AGORATEK International and GEOMATEK, specialise in sampling equipment, geostatistics, QA/QC, due diligence studies, industry training, reference materials and laboratory logistics.



**Toby Day**, Managing Director, Progradex Ltd, United Kingdom

Toby is a mechanical engineer and the owner and managing director of Progradex Ltd. During a 16 year spell in Perth from 1995 to 2011, Toby pioneered the design of the world's first commercially available range of drill sampling systems with Metal Craft in the late 1990s and went on to design the UDR Sampling System in 2004 before both companies were acquired by Sandvik in 2006. Toby founded Progradex Ltd in Perth in March 2009 and developed the new Progradex RC Sampling System and Rig Dust Collectors and has since registered the business in the UK, where he is now based.



**Geoff Lyman**, Director, Materials Sampling Solutions, Australia

### **Novel Derivation of Fundamental Sampling Uncertainty**

Geoff is the principal of Materials Sampling and Consulting Pty Ltd in Southport Queensland. He has consulted in particulate sampling for over 30 years for clients in Australia and overseas. He has worked in the areas of precious metals (Au and PGMs), diamonds, coal, iron ore, sulphides and spent auto catalysts, and grain and meat. He has consulted extensively to Anglo Platinum in South Africa in areas including QA/QC within laboratories, metallurgical accounting at the Waterval Smelter and general advice on statistical problems. His primary interest is providing solutions, both mathematical and practical, to the unsolved problems in materials sampling.



**Kim Esbensen**, Research Professor, Geological Survey of Denmark and Greenland (GEUS), Denmark

### **Theory of Sampling in a Multivariate Perspective: What can chemometrics contribute to Theory of Sampling?**

Kim H. Esbensen, Ph.D, is research professor in Geoscience Data Analysis and Sampling at GEUS (National Geological Surveys of Denmark and Greenland), chemometrics professor with the ACABS research group, Aalborg University, Denmark and external professor (Process Analytical Technologies) at Telemark Institute of Technology, Norway.

A geologist/geochemist/data analyst of training since, since 2001 he has devoted most of his scientific and R and D to the theme of representative sampling of heterogeneous systems and PAT (Process Analytical Technology). He is a member of five international societies. He was chairman of the taskforce responsible for writing the world's first horizontal (matrix-independent) sampling standard (2013) and editor of the magazine TOS forum.



**Francis Pitard**, President, Francis Pitard Sampling Consultants, Canada

### **Sampling Systems for Process Control and Metallurgical Accounting**

Francis has over 40 years of progressive, technical and management experience in the natural resources industry and atomic energy. Accomplished in teaching short courses on the sampling of particulate materials for several Universities and numerous companies around the world, in consulting, in directing the activities of a production oriented research analytical facility with emphasis on innovation and cost effectiveness. Versatile in applying talents in a variety of areas including nuclear chemistry, analytical chemistry, geochemistry, and statistical process control. Outstanding expertise in all aspects of sampling accumulated during a 20 year association with Dr. C.O. Ingamells and Dr. Pierre M. Gy. Dr. Pitard is the author of many papers, three books on sampling and a gold medal recipient from the World Conference on Sampling and Blending.



## Conference Workshop Program

### W1: Concepts in the Sampling of Gold Deposits

Monday 28 July 2014

Snowden is pleased to offer this one-day course offered by Dr Simon Dominy, an expert in the field of gold deposit sampling.

**SNOWDEN**

The course will provide a broad review of the conceptual and practical issues in gold sampling. It has been specially designed for anyone involved, or about to become involved, at the advanced exploration, evaluation and/or exploitation stages of a project. Sampling programmes along the mine value chain aim ultimately to deliver economic tonnes to the mill via the accurate definition of ore and waste. Sampling protocols must be designed to suit the style of gold mineralisation in question.

**This course will cover the following topics:**

- Orebody knowledge and ore characterisation
- Key aspects of the Theory of Sampling
- Critical review of sampling methods available to the geologist
- Designing and implementing sampling programs
- Coarse gold sampling issues

**Cost:** AusIMM member A\$1320 | Non-member A\$1650

**Time:** 8.30 am – 5.00 pm

**Venue:** TBA

**Numbers:** Minimum 6, Maximum 20

**Includes:** Reference notes, refreshments and lunch

**Presenter:** Dr Simon Dominy FAusIMM(CP), Executive Consultant, Snowden Group and Adjunct Professor, WA School of Mines



### W2: Sampling Basics Workshop

Monday 28 July 2014, 8.30 am – 5.00 pm

This practical course covers geological sampling theory and techniques for the minerals industry. It illustrates how sample theory and good sampling techniques are used to control risk and ensure the sampling 'error' is reduced as far as practically possible. Participants will leave the course with an understanding of the importance of good samples, what makes a sample good, an understanding of what sampling and the Theory of Sampling are, and will be able to use the Theory of Sampling to produce a good sampling regime.



**Cost:** AusIMM member A\$1150 | Non-member A\$1400

**Time:** 8.30 am – 5.00 pm

**Venue:** Pan Pacific Perth

**Numbers:** Minimum 6, Maximum 20

**Includes:** Reference notes, refreshments and lunch

**Presenter:** Mark Noppe FAusIMM(CP)  
Principal Consultant, MSc  
(Exploration Geology), Geology



### W3: A Practical Guide to Designing and Running Effective Sampling Programs

Thursday 31 July 2014

This one day hands-on workshop shows you how to design and run effective sampling programs, with all aspects explained using case studies and practical exercises.

**SNOWDEN**

The workshop will cover precision accuracy and bias, sampling errors, sample collection, sub-sampling, and cross-stream sampling. You will:

- Learn how to critically assess and design sampling and sample preparation systems – from drilling through to process sampling

- Understand the importance of good sampling practice
- Understand the sources of sampling error and the cost of poor sampling
- Understand and apply Gy's Theory of Sampling to sample size selection and the design of sampling protocol

**Cost:** AusIMM member A\$1320 | Non-member A\$1650

**Time:** 8.30 am – 5.00 pm

**Venue:** TBA

**Numbers:** Minimum 6, Maximum 20

**Includes:** Reference notes, refreshments and lunch.

**Presenter:** John Graindorge MAusIMM(CP), Principal Consultant – Applied Geosciences BSc (Hons) (Geology), University of Western Australia; Post Graduate Certificate of Geostatistics, Edith Cowan University



To register your attendance please complete the appropriate section of the booking form.

# Sampling 2014 Where it all begins



## General Information

### CONFERENCE VENUE

#### Pan Pacific Perth Hotel

207 Adelaide Terrace  
Perth Western Australia 6000  
Telephone: +61 8 9224 7777  
Email: [reserve.ppper@panpacific.com](mailto:reserve.ppper@panpacific.com)  
Website: [www.panpacific.com/en/Perth/Overview.html](http://www.panpacific.com/en/Perth/Overview.html)



### ACCOMMODATION

The Pan Pacific Perth Hotel is pleased to offer Sampling 2014 delegates a discounted rate of A\$245 per night. To book this conference rate, please contact the hotel directly (details above) and quote AusIMM Sampling 2014.

### EVENT MANAGEMENT

Alison McKenzie, Senior Manager, Events  
Eliza Sanneman, Senior Coordinator, Events  
Belle Doley, Senior Coordinator, Publications

The Australasian Institute of Mining and Metallurgy (The AusIMM)  
PO Box 660, Carlton South, Victoria, Australia 3053  
Telephone: +61 3 9658 6105  
Email: [esanneman@ausimm.com.au](mailto:esanneman@ausimm.com.au)  
Website: [www.ausimm.com](http://www.ausimm.com)

### REGISTRATION DESK

The registration desk will be open:  
Monday 28 July 2014, 4.00 pm – 6.00 pm  
Tuesday 29 July 2014, 7.30 am – 5.00 pm  
Wednesday 30 July 2014, 8.00 am – 3.00 pm

### SOCIAL PROGRAM

#### Networking Hour, Tuesday 29 July 2014

Venue: Pan Pacific Perth Hotel  
Time: 5.00 pm – 6.00 pm  
Cost: Complimentary for all delegates  
Guests: A\$33 per person

#### Conference Dinner, Tuesday 29 July 2014

Venue: Pan Pacific Perth Hotel  
Time: 7.00 pm for 7.30 pm  
Cost: Complimentary for all delegates  
Guests: A\$132 per person

Proudly Sponsored by:

### CONFERENCE PROCEEDINGS

All delegates will receive a printed copy of the conference proceedings containing full papers. Additional copies may be purchased via the registration form and at the registration desk during the conference.

- Additional printed proceedings \$77
- USB proceedings \$55

Proudly Sponsored by:

### NAME TAGS

All participants at the conference will be issued with a name tag upon registration. Your name tag is the official pass to all sessions and must be worn at all times. Lost name tags can be replaced at the registration desk.

Proudly Sponsored by:

### SPECIAL REQUIREMENTS AND DRESS CODE

Every effort is made to ensure people with special requirements are catered for. Should you require any specific assistance or dietary requirements, please include a notation with your registration form to enable us to make your visit a pleasant and comfortable experience. The dress code for the conference, social functions and workshops is smart business casual.

### JUSTIFICATION OF ATTENDANCE LETTER

We know that travel and professional development budgets are tight, and it can be difficult to get approval to attend events and conferences. A justification of attendance letter can be downloaded from the conference website detailing the reasons why attending Sampling 2014 are beneficial for you and your company.

### The AusIMM

- Members of The AusIMM receive a significant discount on the conference and its related activities. This discount is generally above the cost of an individual's annual membership subscription.
- Non-members of The AusIMM receive a great offer relating to AusIMM membership. A letter outlining this offer will be given to you upon registration at the event.
- A selection of conference proceedings, monographs and technical publications will be available to purchase at this conference.
- All attendees receive a full participants list to enhance your networking base.

### Professional Development

It is a requirement of AusIMM membership that individuals engage in an appropriate level of professional development (PD). Maintaining current knowledge and skills through PD activities is imperative to ensuring AusIMM members continue to be the leading professionals in the global minerals sector. Attending or presenting at this conference will contribute towards members' professional development. AusIMM Chartered Professional members and RPEQs can claim 14 hours towards in their logbook.

## PERSONAL DETAILS

Title – Please circle (Prof / Dr / Mr / Mrs / Miss / Ms) \_\_\_\_\_

AusIMM Membership Number (if applicable) .....

AusIMM Membership Credentials (if applicable) .....

Last Name\* .....

First Name\* .....

Middle Name .....

Preferred Name\* .....

Organisation\* .....

Position\* .....

Address\* .....

.....

City\* ..... State\* .....

Post Code\* ..... Country\* .....

Telephone\* ..... Facsimile .....

Mobile .....

Email\* .....

## BILLING ADDRESS (for receipting, if different to the above)

Organisation\* .....

Address\* .....

.....

City\* ..... State .....

Post Code\* ..... Country\* .....

Please tick (✓) if you do NOT wish to appear on the list of participants.

\* Required information

- Please advise any special requirements regarding diet and mobility

## REGISTRATION PROCEDURES

### ATTENDANCE

Only pre-registered, pre-paid registrants will be guaranteed access to the event. Upon receipt of your registration and payment, The AusIMM will send registration confirmation.

### REGISTERING ON-SITE

On-site registrants, with payment only, will be admitted on space availability.

### AusIMM MEMBER RATES

To qualify for the special rates of 'AusIMM member' as quoted on the registration booking form you must be a financial (paid) member. AusIMM 2014 membership fees are due by 1 January 2014. Non-member registration fees apply to all non-members and non-financial AusIMM members.

### METHOD OF PAYMENT – CREDIT CARD ONLY

Payment must accompany all registrations. We accept the following credit cards: Visa, MasterCard, American Express and Diners. For all enquiries regarding payments, please telephone +61 3 9658 6120.

### WHAT'S INCLUDED

We welcome all overseas delegates, members, non-members, authors, new professionals and students to register by completing the registration form and returning it with their remittance to Event Management.

- **Author / delegate / international delegates / non-members / new professionals and student registration includes:** attendance at technical sessions, printed copy of conference proceedings, morning and afternoon teas, lunches, networking and Conference Dinner

- A Student must be currently enrolled full-time at a tertiary institution. Proof of full-time status must be submitted with the registration form.

### PARTICIPANTS LIST

Please note that all registered delegates will have their name, position, company and email address printed in a participants list. Should you not wish to have your details distributed please notify Event Management as soon as possible.

### CONFIRMATION OF BOOKINGS

Conference registrations will be acknowledged as they are received with payment in full. Receipts for registration payments will be attached to the confirmation letter. Please check the confirmation letter and advise of any alterations immediately.

### CANCELLATION POLICY

Cancellations of registration must be in writing only. Refunds will apply as follows:

- More than 28 days before the conference – **full refund**
- 28–7 days before the conference – **refund (less AS\$400 administration charge)**
- 7 days or less before the conference or non-attendance – **no refund (no exceptions)**

An organisation may send an alternative delegate if registration has been paid and the registered person is unable to attend due to unforeseen circumstances. In such cases, the Event Management must be advised of the change prior to the conference.

### WAIVER OF LIABILITY

The AusIMM and CSIRO accept no liability to any persons or body for any loss, injury or damage caused, organised, promoted or sponsored by The AusIMM.

## 1 REGISTRATION Please indicate (✓)

### DELEGATE REGISTRATION

AusIMM member AS\$1265  Non-member AS\$1760  International AS\$1265

COMPLIMENTARY FUNCTIONS – Please indicate if attending (✓)

Networking Hour  Conference Dinner

### AUTHOR REGISTRATION **\*\*All authors must register by Friday 2 May 2014**

AusIMM member AS\$1166  Non-member AS\$1166

COMPLIMENTARY FUNCTIONS – Please indicate if attending (✓)

Networking Hour  Conference Dinner

### SINGLE DAY REGISTRATION (Delegate/Author/New Professional)

AusIMM member AS\$770  Non-member AS\$990  International AS\$770

SELECT CONFERENCE DAY – Please indicate (✓)

Tuesday 29 July  Wednesday 30 July

Complimentary Networking Hour  Conference Dinner at an **additional AS\$132**

### NEW PROFESSIONAL REGISTRATION (31 years & under)

AusIMM member AS\$1166

COMPLIMENTARY FUNCTIONS – Please indicate if attending (✓)

Networking Hour  Conference Dinner

### STUDENT REGISTRATION

AusIMM member AS\$330  Non-member AS\$495

COMPLIMENTARY FUNCTIONS – Please indicate if attending (✓)

Networking Hour  Conference Dinner

## 2 GUEST FUNCTION TICKETS Please indicate (✓)

Networking Hour x ..... tickets @  AS\$33 per guest

Conference Dinner x ..... tickets @  AS\$132 per guest

Guest/s Name(s) .....

## 3 ADDITIONAL CONFERENCE PROCEEDINGS Please indicate (✓)

**Note, that one printed copy of the proceedings is included with your registration.**

YES  I wish to purchase an additional printed copy – AS\$77

YES  I wish to purchase a USB copy – AS\$55

## 4 WORKSHOPS Please indicate (✓)

### W1: Concepts in the Sampling of Gold Deposits

AusIMM member AS\$1320  Non-member AS\$1650

### W2: Sampling Basics Workshop

AusIMM member AS\$1150  Non-member AS\$1400

### W3: A Practical Guide to Designing and Running Effective Sampling Programs

AusIMM member AS\$1320  Non-member AS\$1650

## 5 SPECIAL REQUIREMENTS

Please list any special requirements.

.....

.....

## 6 PAYMENT – TAX INVOICE (INC 10% GST) ABN 59 856 002 494

**PAYMENT MUST ACCOMPANY REGISTRATION – CREDIT CARD ONLY**

Total A\$.....

- **Registration procedures** – Please indicate (✓) that you have read the registration procedures

- **Credit cards** – Please (✓) debit my:

Visa  Mastercard  AMEX  Diners Card

Card No.

Expiry Date: ..... CSV Number: .....

Signature: .....

Please print name of cardholder:

.....

**All enquiries regarding payment, please telephone +61 3 9658 6120**

## How to register: