

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".¹ 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.² 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).³

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.⁴

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".² 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 3077.⁵

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 3077.⁵ 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.⁵⁻⁸ 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 3077⁵ 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 3077.⁵

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 condition- ing — 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 3077⁵). 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.² This inference by the guide is very much correct, see DS 3077 2013,⁵ 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

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”,⁹ 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



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

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.¹⁰

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.¹¹ As correctly stated in the grain guide “it is important to ensure, as far as possible (*s/c*), that all grains in the aggregate have an equal chance of being included in any sub-sample drawn from it”.² 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.

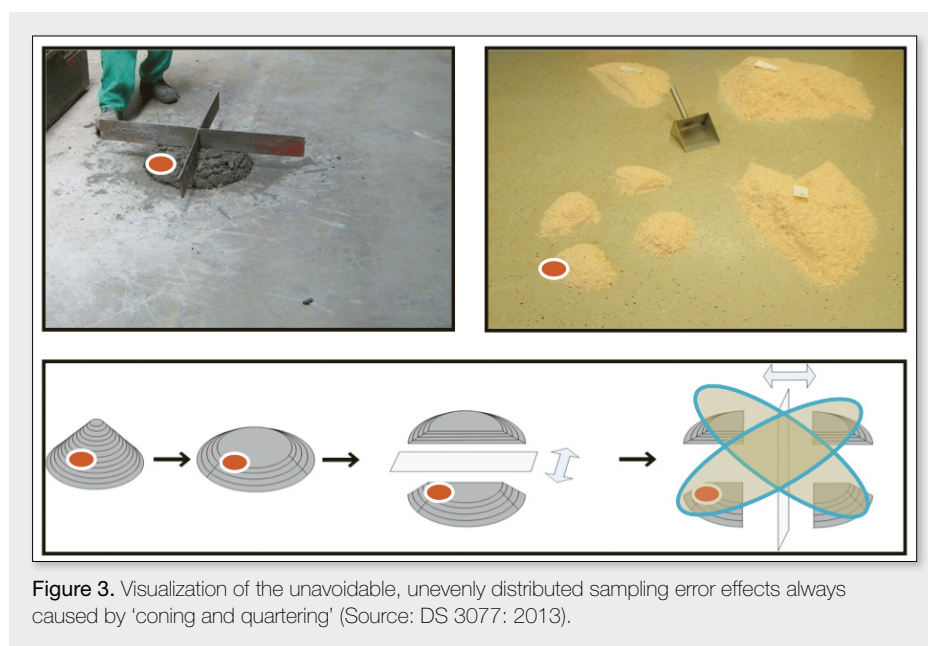


Figure 3. Visualization of the unavoidable, unevenly distributed sampling error effects always caused by ‘coning and quartering’ (Source: DS 3077: 2013).

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.¹² 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.⁴ 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.⁴ 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,^{13–15} 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*.