Development and Evaluation of Efficient Field Deployable Sample Preparation: Fit-For-Purpose Representative pXRF

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ABSTRACT

Since acquiring the designs of the REFLEX™ Instruments Sample Preparation range in early 2024, Block10 has embarked on an ambitious redesign, updating the high-quality crusher, mill and press equipment to meet the evolving and growing interest for small, portable, field deployable analysis solutions in the mining and exploration industries. This article outlines the features and benefits of this expanding product range, presenting Replication Experiments (TOS) performance evaluation and other test-work to highlight the high precision, low bias, highly effective safe operating potential of Block10 preparation equipment for pXRF and other analytical modes.

1. Introduction

When analysing geological samples three things matter: quality, cost and turn-around-time. High quality central laboratories are located in most developed resource regions in the world, and on-site at larger mining operations. These laboratories are capital intensive, with high investment required for permanent equipment, personnel and processes. Such operations require large sample quantities and a continuity of work in order to achieve a reasonable unit cost.

The scale of these laboratories, often processing thousands of samples daily, means that they are invariably located in the larger and more established locations. Those working further afield – the explorers, drilling sites and junior operations - must send their samples over great distances (and at great expense) and wait weeks or even months for assay results. The cost of shipping and submitting samples, whilst significant, is often dwarfed in comparison to the opportunity cost of slow results; processes run sub-optimally for longer, and sampling resources cannot be targeted to greatest effect. But results need not be "online" or "real-time" in most instances; the value is unlocked in having confidence in turn-around-times measured in hours, not months.

There are many examples of mobile sample laboratories being successfully deployed into remote locations to combat this tyranny of distance, but the scale of these "mobile" solutions is usually based on shipping container multiples, and that is for the sample preparation equipment alone.

The introduction and widespread availability of portable XRF analysis (pXRF) instruments has opened new opportunities to address these challenges. By 2001 the technology had advanced to a level to make lightweight, safe, reliable, accurate instruments a reality, and since then, their application and use has proliferated, with multielement analysis on a sample possible in minutes. Most recently, the patented detectORE™ process (Portable PPB, 2024) has expanded the applicability of pXRF to attain low level gold results down to low parts per billion (ppb) levels. Portable-XRF has also proved popular within the larger laboratories, as an efficient, cost-effective multielement analysis solution and for initial screening purposes.

Recognising the opportunity for pXRF, and seeing very limited solutions in the market, REFLEX™ Instruments, a leading IMDEX brand, developed a crusher, disc mill and sample puck press for the optimal preparation of samples for pXRF analysis.

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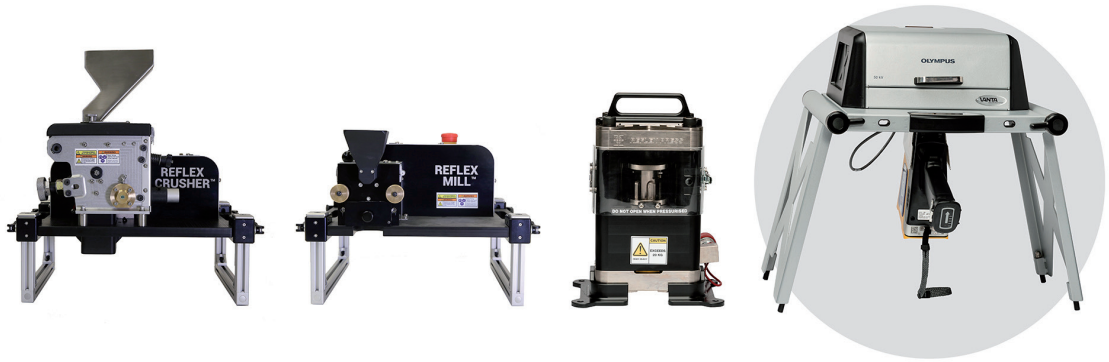


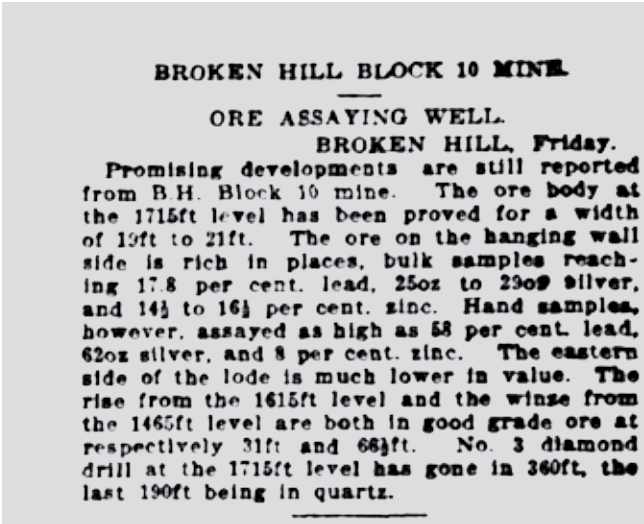
Figure 1: The range of REFLEX Instruments.

The attributes of reliable operation, compact size and high-quality output make this product suite highly applicable to other analytical techniques too; their use is by no means limited to pXRF.

In early 2024, Block10, a company specialising in the development of automation, sensing and mechanical solutions for the mining industry, acquired the designs as the new vendor of the former REFLEX-Crusher, REFLEC-Mill and REFLEX-Press in-field preparation tools (IMDEX, 2024). In consultation with existing clients and users, a design review and update has been undertaken, culminating in the release of the new BX-C Crusher and BX-M Mill.

Leveraging the latest in motor and manufacturing advancements, these new releases offer significant equipment weight and size reduction, improved safety, many optional customisations, and high-quality results. The product range has also been expanded to include the BX-R Riffle Splitter, and further developments are ongoing to meet the specific requirements of the industry into the future.

A comprehensive evaluation of the Block10 equipment performance has been undertaken, with results highlighting the powerful sample preparation potential, whether in-field, remote, or at central laboratories.



BROKEN HILL BLOCK 10 MINE.

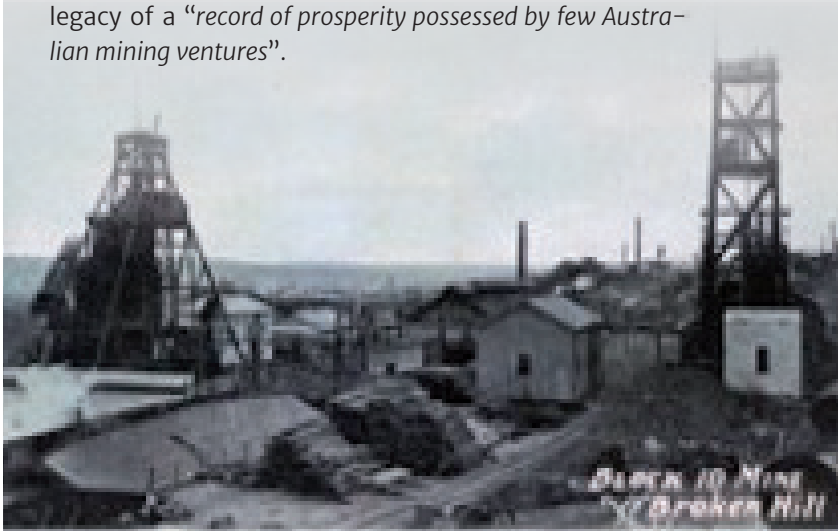
ORE ASSAYING WELL.

BROKEN HILL, Friday.

Promising developments are still reported from B.H. Block 10 mine. The ore body at the 1715ft level has been proved for a width of 19ft to 21ft. The ore on the hanging wall side is rich in places, bulk samples reaching 17.8 per cent. lead, 25oz to 29oz silver, and 14½ to 16½ per cent. zinc. Hand samples, however, assayed as high as 58 per cent. lead, 62oz silver, and 8 per cent. zinc. The eastern side of the lode is much lower in value. The rise from the 1615ft level and the winze from the 1465ft level are both in good grade ore at respectively 31ft and 68½ft. No. 3 diamond drill at the 1715ft level has gone in 360ft, the last 190ft being in quartz.

The BHP Block 10 Co. Ltd was floated on March 14, 1888 and struck ore in December 1889. The mine went on to produce over 40 million oz of Silver, 400,000 tons of Lead and 400,000 tons of Zinc over a 35 year life. With declining grades and a collapse in metal prices following the end of World War I, Block 10 was liquidated in 1924, and its mine purchased by the Broken Hill Proprietory Company Ltd (BHP).

A century later in 2022, Block 10 Pty Ltd was established by decendents of these early mining pioneers, with the spirit of innovation and enginuity continuing the legacy of a "*record of prosperity possessed by few Australian mining ventures*".



2. Block10 Sample Preparation Methodology

Although extending its involvement upstream to sample collection on specific projects, the Block10 Sample Preparation Methodology typically begins with a sample, extracted previously by others and presented in a calico bag or similar.

The specific primary sampling method – reverse circulation (RC) drill chips, diamond core, manual or mechanical grab or auger samples, cross-belt or falling-stream process samples – and the relative merits of these methods (Theory of Sampling (TOS)), is not considered here; the focus is on accurately preparing and representing these samples for analysis in the field.
Optimal presentation usually involves pressing a finely milled sub-sample into a 30mm puck, with the high hydraulic force and quality dies resulting in a smooth surfaced, well-mixed sample. Whereas higher homogeneity is possible by fusing a bead (for example, with an xrFuse Electric Fusion Machine (XRF Scientific, 2024)), the requirements for high temperature (1200°C) operation, expensive platinum crucibles, and additional flux dosing means that a pressed puck can reasonably offer an optimal balance between portability, performance and cost. Studies (Rohiman & Arifin, 2020) have also validated that pressed samples are superior for trace element (<100ppm) analysis, where fused beads are impacted by high (flux) dilution.

The Block10 general methodology for sample preparation for multi-element analysis can be summarised as:

a) Obtaining a representative sample (typically >=500g), with particle size <30mm (TOS to the fore).
b) Crushing to <2mm in the BX-C crusher.
c) Dividing to 250g, then 125g in the BX-R riffle splitter.
d) Milling to <100µm in a BX-M mill.
e) Riffle splitting again to (a nominal) 62.5g.
f) Scooping 10mL into a sample die. Some samples may require the addition of a binder.
g) Pressing the sample in the BX-P hydraulic sample press.

The samples may then be analysed with a pXRF instrument, or other techniques.

Many variations to this standard methodology exist, and in each application the steps, settings and processes can be adapted to best suit the specific requirements.

For example:

- Some fine samples may not require crushing and can be milled directly.
- Some samples containing heavier elements at macro levels may be analysed as a milled powder rather than a pressed puck.
- Different sample materials may require crushing & grinding at smaller size to achieve homogeneity or may tolerate a wider size for faster operation.

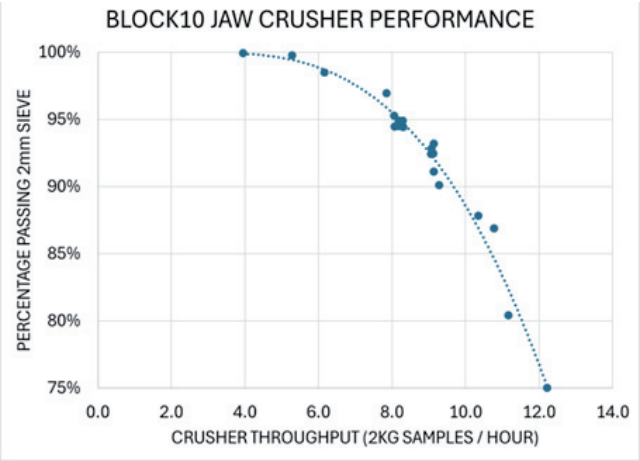
Credit: Block10; used with permission.

Figure 2: The updated Block10 BX-C Crusher and BX-M Mill

3. Crushing and Milling Specifications

Regarding crushing and milling specifications, typical targets are 90% passing 2mm and 90% passing 100µm respectively. These are the settings used for the testwork included in this article. Adjusting these settings is a quick process using a supplied hand tool to turn an adjusting screw.

In selecting an appropriate crushing specification, the throughput of the machine should be considered as one of the competing objectives: finer crushing to smaller particles (higher percentage passing 2mm) vs faster processing. Several samples of "bluemetal" (<20mm screened basalt) were tested with varied jaw gap adjustments, to plot the curve in Fig. 3, showing a highly productive ~10 samples / hour if processing 2kg samples at a 90% passing 2mm specification. These performance curves are dependent on material type and can also be impacted by other factors (e.g. sample moisture content), so results will vary for different sample types.



Credit: Block10; used with permission.

Figure 3: Block10 crusher throughput performance

Sizing specifications require routine validation as part of the QA/QC process, most simply and reliably achieved by sieving a sub-sample in the applicable sieve size. For crushed size specification, dry sieving suffices, unless there are high concentrations of agglomerating fines present, whereas wet sieving is required to accurately determine the mill specification. There are other alternatives such as laser or vision-based particle size analysers.

The crushing stage takes the sample to a 2mm top-size. Portable XRF analysers typically have a FoV spot size between 3mm and 8mm (MicroXRF, 2024), so in most cases it is best practice to further reduce the particle size before analysis to present a better mixed sample.

Samples are typically milled to a range between <50µm and <200µm specification, with ~75-100µm preferred.

- If particles are too large, they may not bind together properly when pressed, and pucks simply crumble.
- Larger particles closer to the sample surface can also "shadow" smaller particles behind them which may then not be quantified properly.
- Lighter elements (e.g. Na, Mg, Al, Si) are only detectable at shallow depths with low energy X-rays"; Na for example can only be analysed in the first ~10µm of sample. When analysing for these lighter elements the impact of surface roughness and particle size is much more pronounced than for heavier elements (e.g. Fe, Cu), where larger penetration depths make for less susceptibility to particle size influence.

4. Machine Safety

Electrical equipment (e.g., the crusher and mill) are fitted with emergency stop circuits, and sensor interlocks to prevent operation whilst the covers are open, or sample trays/chutes are missing. The electrical enclosure is fitted with tamper resistant fasteners.

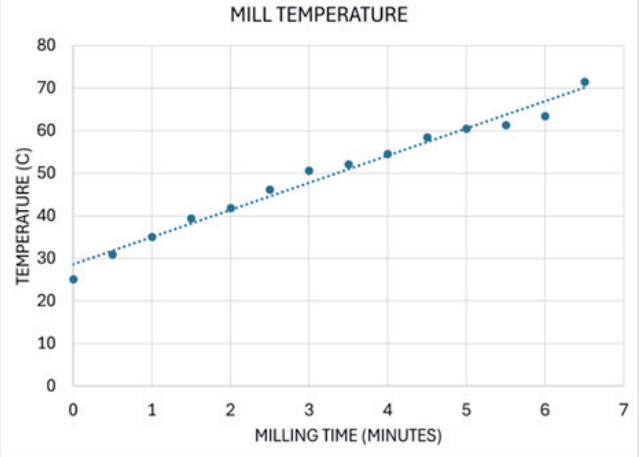
The updated BX-C and BX-M equipment features dual redundancy safety, to give improved safeguarding of the machinery to SIL 2 (IEC 61508) / Category 3 (IEC 60204-1). The use of a safety relay allows detection of short circuits and any lack of simultaneity between the two channels trips the emergency stop.

5. Milling Specification – Thermal Impacts

Whilst a lower milling specification (in terms of output particle size) is often desirable, it requires longer milling times, and the corresponding increase in duty cycle causes an increase in temperature.

Unlike a conventional ring or puck mill, where the bowl is enclosed and vibrating, the Block10 BX-M mill, with its rotating disc design allows much better temperature dissipation, via aluminium heatsinks that draw heat away from the milling disks. Fig. 4 shows a typical temperature gradient for a milled sample.

Geological samples are typically dried at 105°C, so should suffer no ill effects even if milled for extended times at temperatures below this, however if temperature sensitive samples are processed, consideration should be given to detecting and limiting the temperature.



Credit: Block10; used with permission.

Figure 4: Milling temperature.

Block10 offers several upgrade options including:

- Temperature sensor for monitoring,
- Temperature controls, to regulate operation and inhibit milling if temperatures exceed a threshold,
- Water Cooling.

Ultimately, the best solution is to limit the heat generation in the first place, either by a relaxed milling specification or smaller samples (both reduce the milling time and duty cycle), or with multiple mills if one unit is being over-utilised.

6. Dust Control

Dust control (or lack thereof) has dual impacts on personnel safety and sample quality.

From a quality perspective, higher extraction airflow is not necessarily better, as it removes sample material (particularly fines) and biases the result. However, too low an airflow increases the likelihood that material will build up and carry over between uses, contaminating subsequent samples.

The key to optimal extraction is to focus on fugitive dust only, leaving particles that may have become airborne but still within the sample chute / jaws / trays every opportunity to remain as part of the sample flow. Only once dust exits the vessel should it be extracted away (as this sample material was lost to the process anyway, so dust extraction does not further bias the results).

The Block10 BX-D dust extraction system is configured to give operational flexibility with one, two or three machines and includes a HEPA-14 filtration system, reducing airborne contamination and improving cleanliness.

The HEPA-14 filter (to EN 1822:2019) captures at least 99.995% of particles 0.3µm or larger. It does not remove the need for personnel to wear appropriate PPE (including respiratory protection), in accordance with local requirements, but it does give added protection and reduced contamination.

Samples by definition contain unknown components, and with an ever-increasing understanding of the risk of dust exposure (silica, asbestos) it is becoming increasingly critical to manage and suppress dust generation in all workplaces. Whilst central laboratories typically have ducted dust extraction systems, the remote/field operations often lack this infrastructure, so the BX-D equipment offers an efficient, lightweight and portable means to address dust.

The inclusion of a HEPA-14 filter ensures that harmful particles are efficiently and safely captured - rather than being recirculated into the environment as is often the case with other vacuum systems.

7. Contamination

The typical sample loss in the Block10 crushers are <0.5% by mass, with effective dust control in place. Only a portion of this is carried over to the next sample, with the balance extracted or escaping as airborne dust. The crusher infeed chute can be removed via a quick release to give quick access to the crusher jaws between samples for visual inspection, and if necessary, an additional vacuum or brush if there is visible carryover material remaining in the jaws.

The Block10 mills are also readily cleanable and can be opened in seconds without tools. Best practice is to open the top of the mill every cycle and brush any residue into the outfeed bin to ensure it remains with the sample. Once the sample bin is removed, a more aggressive suction and brushing, including for the inside of the milling disks, ensures any contamination to the next sample is minimised. Following this 'good laboratory practice', with dust extraction, milling losses are typically <1%.

Figure 5: Cleaning the crusher between samples

The other potential source of contamination is from the active wear parts – the jaw plates in the crusher and the discs in the mill. These parts wear with use, and the lost material ends up as a contaminant in one and more samples.

Block10 crushers and mills feature full tungsten carbide wear parts, for ultimate hard-wearing performance. Hardened steel wear parts are available if tungsten (W) is an analyte of interest.

Most soft(er) samples may not perceptibly wear the tungsten surface, however tests milling a highly abrasive, high surface area graded silica sand (0.6–0.8mm) did indicate W contamination, at low but detectable levels. Such results were not observed when milling other reference materials. In the vast majority of cases, W contamination is either negligible or of no significant interest, so tungsten carbide offers best performance, with optimal lifetime.

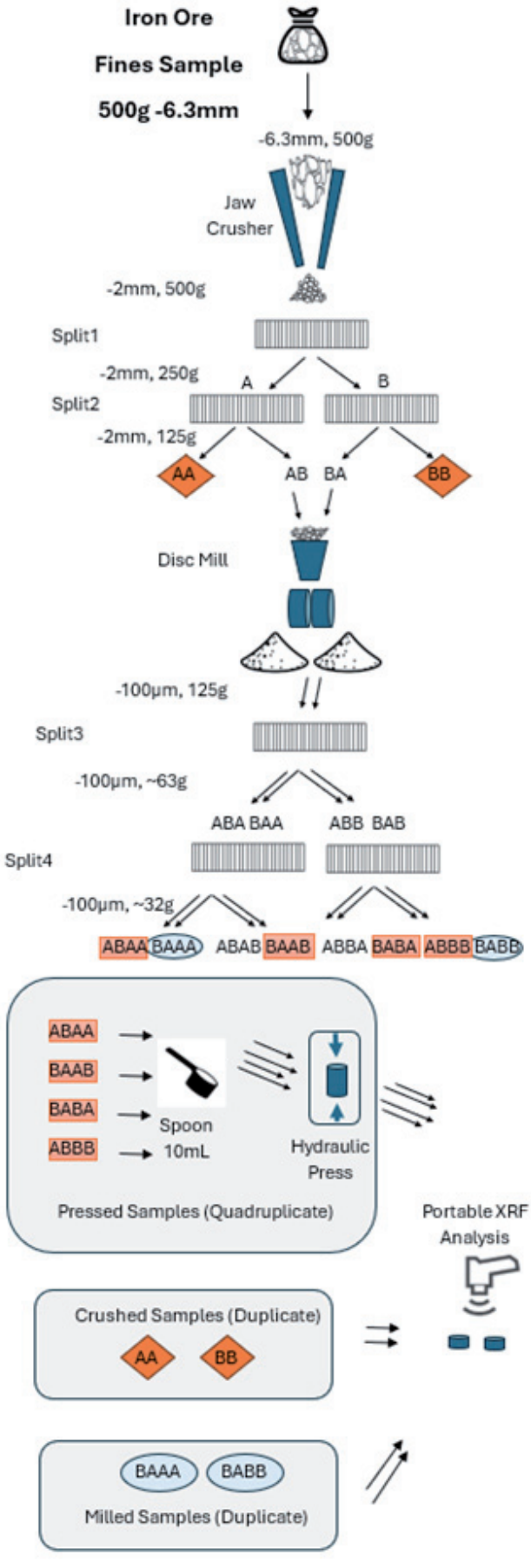
8. Example: Iron Ore Fines Sample

As an illustration of the effects of crushing, milling and pressing, an iron ore fines sample (<6.3mm top-size) was processed and analysed with an Evident Vanta™ Handheld XRF Analyzer, following the methodology outlined above to obtain quadruplicate pressed pucks. Separate splits were also taken at different stages of the full field sample preparation pathway, (see Fig. 6):

- Three "grab" samples with 10mL scoops were taken directly from the sample bag and analysed without further preparation.
- Duplicate (riffle split) samples analysed after first crushing to 2mm top size.
- Duplicate (riffle split) samples were analysed after milling to 100µm top size.

For analysis of loose powders, a cup with polypropylene or mylar film bottom is typically used to present a uniform 'flat' sample surface which is then analysed from below with an upwards projecting pXRF instrument. Loose powder samples do not have the same sample packing density and surface smoothness as a pressed puck but can be simpler to prepare (notwithstanding the assembly of the consumable cup and film can be fiddly, time consuming and costly).

The relative sampling + analysis variability (RSV), or Relative Standard Deviation (RSD), also known as the Coefficient of Variation (CV), is defined as the ratio between the standard deviation (σ) and the mean (µ), RSV = σ / µ of a replicated sampling or sub-sampling operation + analysis. As a dimensionless measure (i.e. expressed as a percentage) it allows for effective comparison of the precision between assays as a function of different sub-sampling operations.



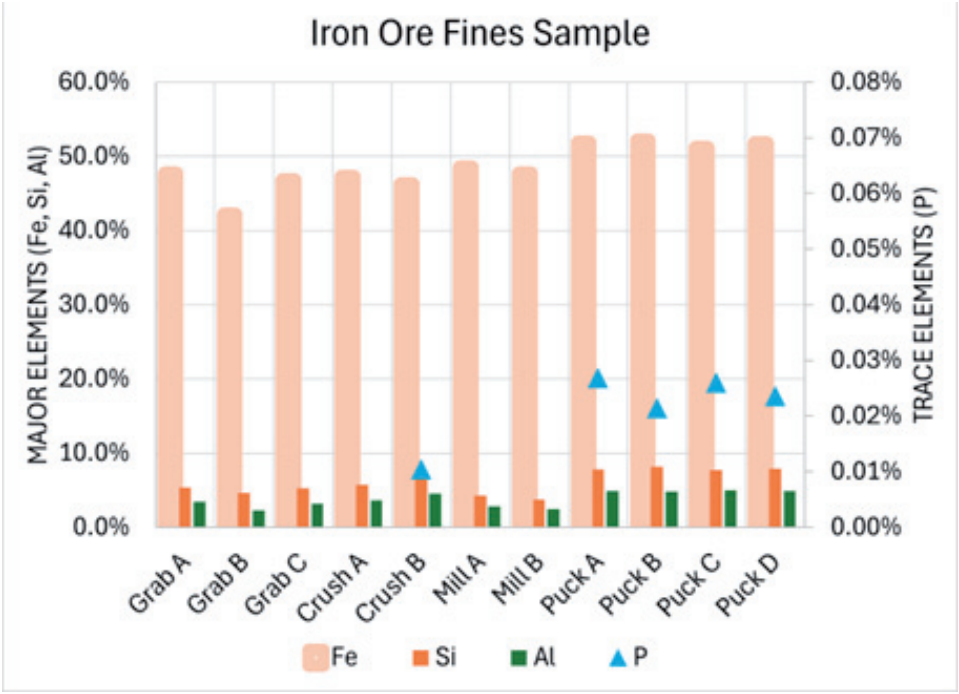
Credit: Block10; used with permission.

Figure 6: Process flow for iron ore sample preparation evaluation

Although in this limited first evaluation the data sets are small (more expansive testing will be performed in future), it is nonetheless illustrative in highlighting the benefits of well-executed sample preparation in the field. The RSV% is calculated across four main elements of interest in the iron ore sample, the valuable element being iron (Fe), and the deleterious aluminium (Al), silicon (Si) (as silica) and phosphorus (P), as presented in Fig. 7 (concentration) and Tab. 1 (RSV%).

The assay precision for Fe, a heavier element existing in high grades (~50%), improves with additional sample preparation (crushing, milling, pressing), but only incrementally; if the only concern was for an Fe result, it may be perfectly acceptable to stop at crushing, or analyse the fines sample at its raw size (<6.3mm).

Deleterious elements are also of significant interest, since they can have a negative impact on the produced ore value and must therefore also be quantified. For Al and Si, both much lighter / lower energy elements and hence more susceptible to sample surface roughness, there is a marked improvement (reduction) in RSV% when the sample is pressed into a puck, Table 1. The most striking example of the benefits of pressed pucks concerns measuring phosphorus, P. As a light and low (trace) concentration element it fails to be detected at all for most of the preceding sample preparation steps; it is only after being pressed into a pellet that it can be measured reliably.



Credit: Block10; used with permission.

Figure 7: Iron ore sample prep + analysis comparisons.

Table 1: RSV% for replicated prep + analysis.

Sample Prep	n	RSV (Fe)	RSV (Al)	RSV (Si)	RSV (P)
Grab	3	6.5%	17.7%	6.3%	~
Crushed	2	1.6%	11.1%	8.2%	~
Milled	2	1.2%	8.2%	6.3%	~
Pressed	4	0.81%	1.4%	2.2%	8.7%

9. Pressed Puck Analysis

For sample analysis, pressed pucks provide superior results. The Block10 BX-P press can be used with two different dies:

- the standard die produces ~26mm diameter pucks from the sample material only, and typically have sufficient integrity to allow analysis, but may chip around the edges or crack during excessive transportation, handling, storage, etc.
- For longer lasting pucks the premium die may be used, where a plastic retaining ring (and optional caps) are used.

Credit: Block10; used with permission.

Figure 8: Block10 sample press dies.

'Infinite Thickness' is the minimum thickness a sample must have in order to absorb all the x-rays of the primary X-ray beam emitted from an XRF instrument. If the infinite thickness is not met, then some of the x-rays pass through and are lost from the sample and result in underreporting of some elements. (Portable Spectral Services, 2024) The eventual thickness of the pressed pucks varies with the compressibility of the sample material, but typically a 10mL volume of freshly milled sample is suitable for creating a well-formed puck, with sufficient thickness.

10. Binders

Many sample types will bond into a puck easily, under hydraulic pressure (Block10 uses 19 tonnes of force in producing 26mm pucks), however some will fail and further additives are required:

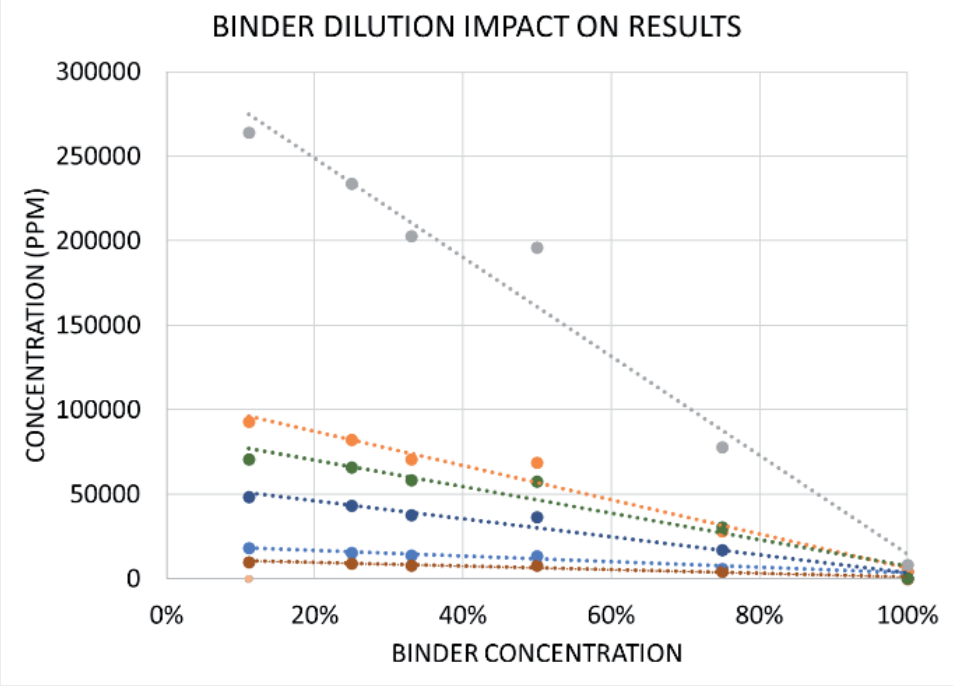
- For hard materials like high quartz samples, the puck may not bond properly without the addition of a wax / cellulose binder. Block10 produces a binder that is typically used in dilutions of between 3% and 12%; the binder is placed in a mixing vessel with 10mL of milled sample prior to puck pressing.
- Less commonly, samples that may fluidise under pressure (e.g. higher clay content) may also fail to form a puck; rather than bind into a solid, the sample behaves like a liquid and is simply forced out of the (small) clearances in the die components. In such cases some "roughage" may need to be added to the sample to ameliorate this fludising propensity; a milled bluemetal (basalt) or feldspar material added in ~25% dilution will usually suffice. Alternatively, the sample may be pressed at a lower pressure.

Whenever a sample is diluted with binder material, it is important that both the volume and composition of the binder are known, so that a correct compensation may be applied to the results.

The composition of the Block10 supplied wax binder is shown in Tab. 2. With a typical general composition of CnH2n+2, the bulk of the binder (~98%) consists of light elements that are outside the focus of pXRF mineralogical analysis (typically starting at Mg on the periodic table).

Table 2: Block10 binder chemical composition

Element	Concentration
Mg	0.42%
Al	0.45%
Si	0.84%
P	0.0070% (70ppm)
K	0.030%
Ca	0.050%
Ti	0.010%
Mn	0.0015% (15ppm)
Fe	0.030%
Cu	0.0003% (3ppm)
Zn	0.0004% (4ppm)
Th	0.0039% (39ppm)
U	0.0014% (14ppm)



Credit: Block10; used with permission.

Figure 9: Binder dilution impact.

Diluting the sample with a binder will be apparent in the results; however, for many samples in the grade ranges of interest, these low-level concentrations can be considered to have negligible impact on the results. The binder material also makes for a good reference blank, but it is challenging to prepare a puck at 100% concentration. Block10 can supply such reference blanks, although not yet as a certified reference material (certification is likely in the near future).

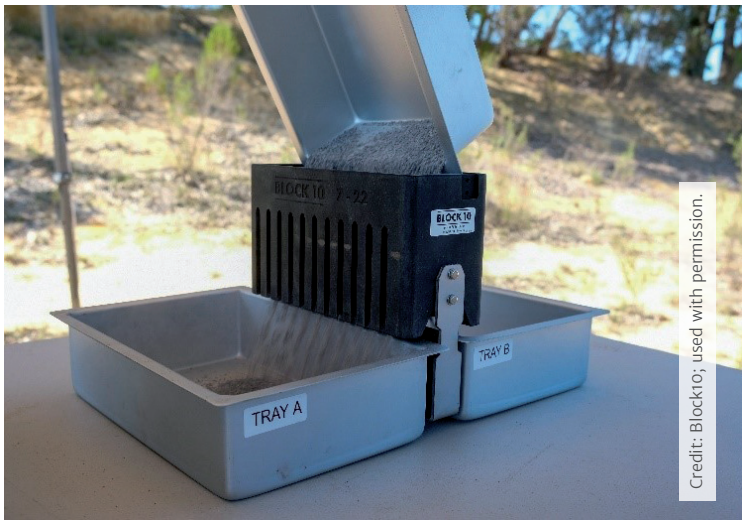
Even if assumed blank, the dilution impact of the added binder should be considered. At low level dilutions (e.g. 6%, as used for many of the samples in this testwork) the impacts are minimal, and where the focus is comparative rather than absolute results, may be disregarded, but at elevated levels (only used if necessary, i.e. previous attempts to form a puck with less binder have failed) the grades can be corrected (raised) proportionate to the dilution ratio.

Fig. 9 illustrates the impact on measured concentrations (highest to lowest Si, Al, Fe, Ca, Mg, Ti) for pressed pucks prepared from IMS-393 certified reference material and diluted with between 12% and 100% binder.

11. The Block10 BX-R Riffle Splitter

As the latest addition to the sample preparation range, the BX-R riffle splitter was developed to address the several sample division stages typically used when preparing samples with the rest of the Block 10 product range.

The BX-R riffle splitter features 13 chutes on each side for a 50:50 split. The chutes are 7mm wide to give >3x the maximum particle size after crushing (2mm) to prevent clogging. Although enclosed riffle splitters designs have been recommended as good practice (Esbensen & Wagner), the Block10 design is open, to allow constant visual inspection of the flowing particles. This is a more reliable configuration that limits the risk of internal sample hangup and carryover between samples. However, if an enclosed specification is required then a hood option is also available.



Credit: Block10; used with permission.

Figure 10: Block10 field deployable BX-R riffle splitter.

The splitter is machined from a single piece of high lubricity plastic. By machining rather than adopting the fabrication processes (bending, cutting, welding) used in most other rifflers, the geometry of the chute spacing can be much more tightly controlled, and a perfectly uniform chute width is key to the performance. The plastic construction means that it is very lightweight and can pack into its carry case along with size-matched lightweight aluminium trays for a highly portable splitting solution.

Splitting equipment is judged on two key criteria: performance precision and bias.

A. Khan's much-cited thesis (Khan, 1968) compared sample division methods, and concluded that rotary sample division was optimal, followed by riffle splitters. Khan considered a single sample mixture (60% fine / 40% coarse sand), calculating a standard deviation of 0.125% for rotary and 1.01% for chute riffling. A similar methodology using a 1:2 mixture of iron chips to sand, was conducted more recently (Nenuwa, Oke, & Sanya, 2018), with much less favourable rotary division performance; with standard deviation of ~2% and RSV of ~4%.

The Block 10 Portable Field Sample Preparation Equipment for pXRF is destined for extensive further assessment, planned to cover a wide range of rock types and relevant operating conditions. Systematic DOE (Design of Experiments) will be used.

Specifically, to evaluate the riffle splitting equipment at this point in time a test was devised utilising a binary mixture of white rice and chia seeds. Both are free flowing materials, but with a significant size disparity (see Fig. 11) that will cause segregation, and which also allow for easy separation by sieving. Both rice and chia seeds have good integrity and do not crumble easily, an important attribute for replication testing involving re-mixing of the original material batches.

This test was conducted at chia seed concentrations of 1%, 5%, 10%, 20%, 50%. Sets of replicated 4-fold sample divisions were performed at each concentration, with the component parts re-mixed completely after weighing the rice and chia fractions of the LH and RH splits (simulating 'analysis' of component concentrations).

Conceptually, a perfect single particle split should have a mean of 0.5 and an RSV of 100%, whereas when the number of particles approaches infinity, the RSV% should decrease asymptotically to zero.

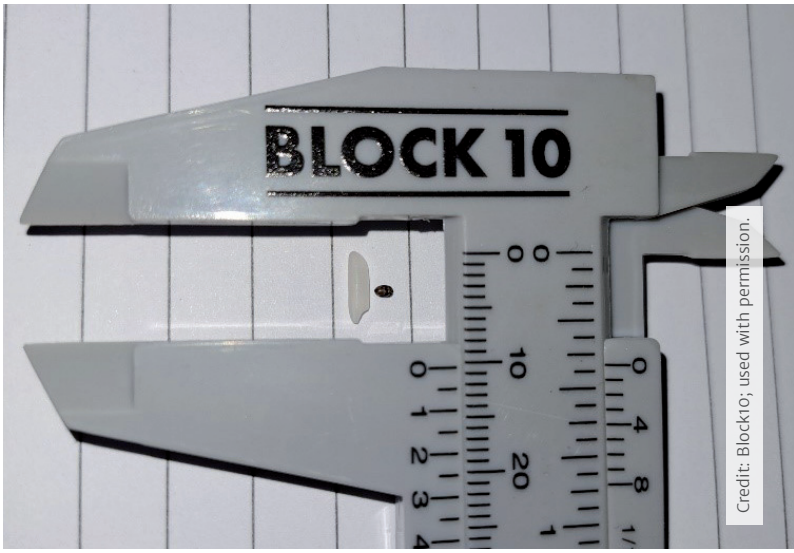


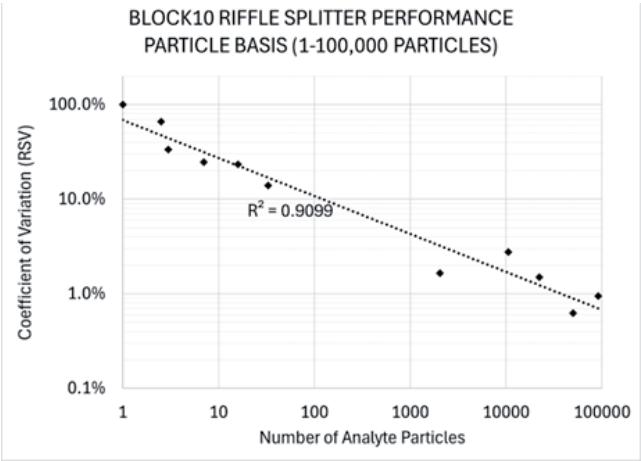
Figure 11: Size comparison for rice and chia seeds.

However, in practice, sub-sampling errors and measurement uncertainties, will cause variability in the RSV% results.

At extremely low particle counts, all the way down to the trivial case of sample division of a single particle, a number of rice grains (1, 2, 5, 8, 16, 32) were counted and put into 90 grams of chia, then divided with the 50/50 BX-R riffle splitter (again replicated four-fold). Why should consideration be given to such low particle counts? For many commodities at major and minor grades it is not relevant, but in a case such as gold analysis, where typical fire assay aliquots of 30g are prepared at 75µm top-size for grades that can be below 1ppm, there really aren't many analyte particles to split! Samples with coarse gold where most of the particles are at or near the milling specification have few Au particles in the aliquot. In this very challenging range, performance results indicate a likely RSV of 15-25% for the BX-R splitter.

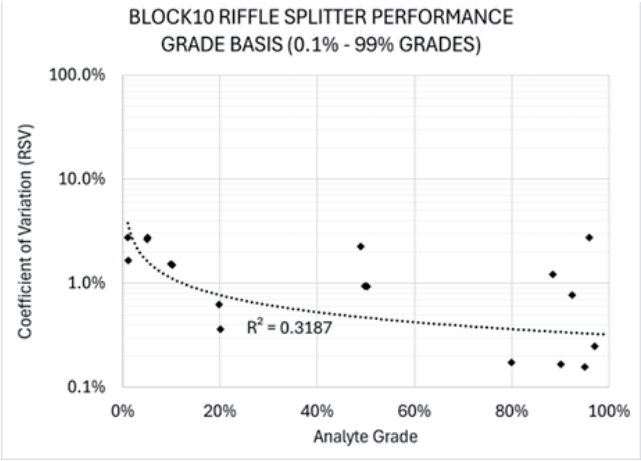
The precision results are plotted in Fig. 12 and Fig. 13, depicting the predicted trend in practice – a 100% RSV at a particle count of 1, improving to a sub 1% RSV for analyte grades >20%.

The splitter performance is likely to differ for different materials, and will be impacted by particle shapes, flow properties, etc. However, the following of the theoretical trend, and the RSVs lower than 3% across all tested major grades suggests the BX-R riffle splitter produces high quality, precise results.



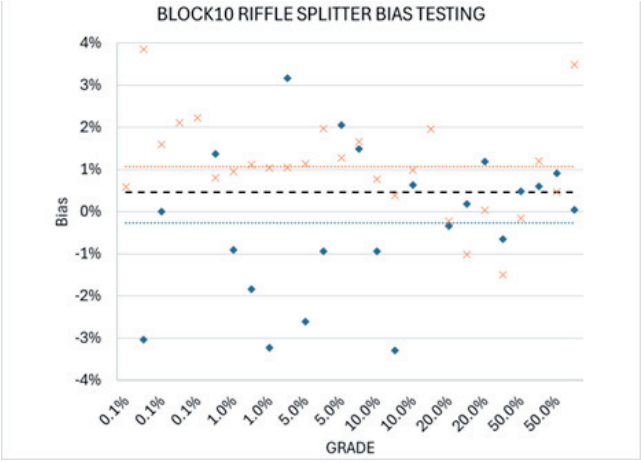
Credit: Block10; used with permission.

Figure 12: Riffle splitter evaluation.



Credit: Block10; used with permission.

Figure 13: Riffle splitter evaluation.



Credit: Block10; used with permission.

Figure 14: Riffle splitter bias test.

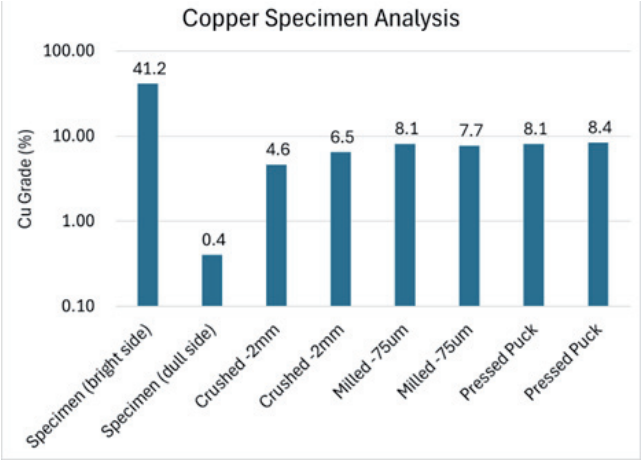
LH vs RH split bias is plotted in Fig. 14, and depicts an average rice (orange crosses) bias of +1.07%, an average chia seed (blue dots) bias of -0.26%, with a combined bias across both analytes of 0.4%. The bias is mainly attributable to the geometry of the splitter, and any slight variations in the flow, particularly of the last-most chute on either side, will impact this balance. This is where a rotary divider whould outperform a stationary splitter, with the potential for much greater than 13 sub-divisions making up each split. However, for the trade-off with size, weight, cost and complexity that is required for a portable and field-deployable solution, this is a welcome quality result.

Further test-work could include similar evaluation of a range of different splitter designs and configurations. It would be most useful across a range of equipment manufacturers to produce similar precision performance curves, allowing evaluation that the splitting device (and the corresponding KPIs) are fit for purpose at a given grade.

12. Example: Cu Specimen Analysis

Similar to the iron ore fines sample, a single azurite (copper) specimen rock, weighing approximately 100g, was also considered.

When in the field, coming upon an interesting sample, the immediate temptation when armed with a pXRF instrument is to analyse. This is fine for identification purposes, as long as no inference is made on grade based on these data.



Credit: Block10; used with permission.

Figure 15: Copper specimen analysis example.

Credit: Block10; used with permission.

Figure 16: Copper specimen, crushed chips, puck.

Table 3: RSV% comparison of crushed, milled and pressed sample

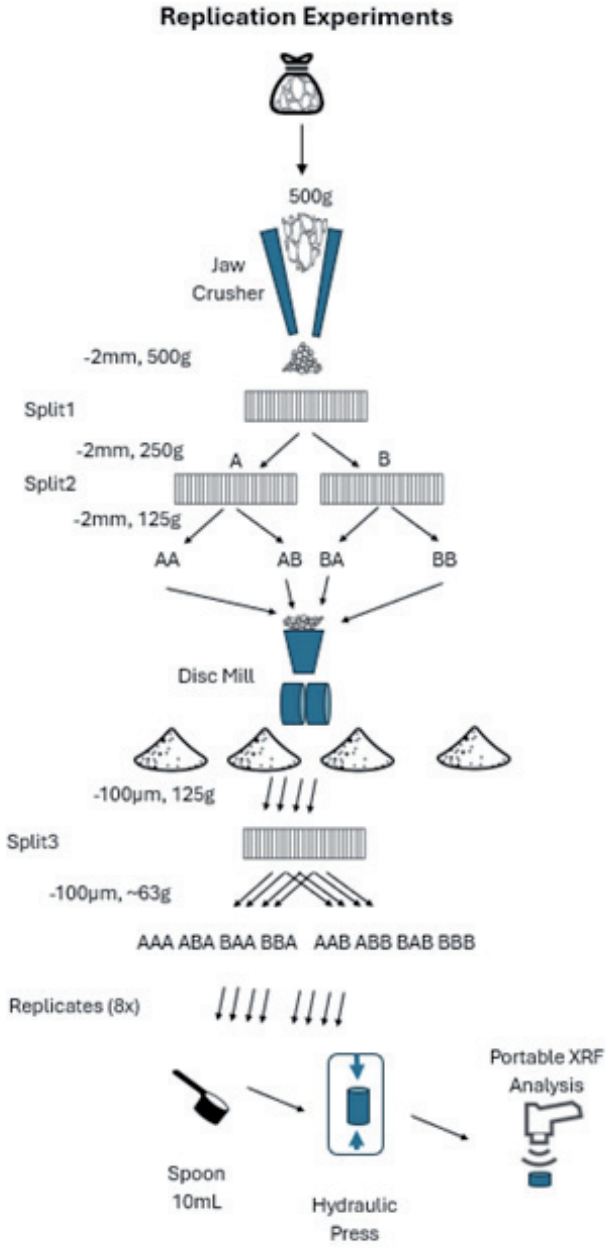
Sample Prep	n	RSV
Cu Specimen Only	2	98.1%
Crushed (-2mm)	2	16.5%
Milled (-75um)	2	2.6%
Pressed Puck	2	2.0%

As shown in Fig. 15, the results will unsurprisingly vary wildly depending on whether the interesting (blue) side of the rock or the barren back is facing towards the pXRF, while duplicate measurements of crushed, milled and pressed stages again highlight an improved RSV% with further sample preparation. There is still considerable variability at crushed (2mm) size chips (clearly visibly evident in Fig. 16), and interestingly, at these grades (~8% Cu) there is only a marginal improvement between the milled and pressed samples.

In reality, copper deposits have much lower sub-percentage cut-off grades, where the difference between milled powder and pressed pucks will likely be much more pronounced. Pressed puck preparation is highly recommended for more precise, less variable analysis.

13. Replication Experiments

In the domain of sample preparation (rather than primary sample collection, which should be considered separately) a Replication Experiment (TOS) was devised to provide insight into the variability (precision) of the final analytical results, when using the present Field Sample Preparation Methodology and analysing all 8 split pathways (see below). The complete subsampling pathway is shown diagrammatically in Fig. 17.



Credit: Block10; used with permission.

Figure 17: Replication Experiments process flow.

The Replication Experiment was conducted on two very different sample types: a Certified Reference Material (CRM) vs. a field collected mineralized sample:

- a 500g CrushedCRM™ produced by IM Standards (IMS-393).
- a 500g Arsenopyritic / Quartz sample from the WA Goldfields region.

The CrushedCRM™ is designed with inherent heterogeneity, to allow sample preparation processes to be assessed with a reference material. Reference materials introduced at the pulp (milled) stage only validate the analysis rather than the preparation.

A good replication result (low RSV% across 8 splits, across each element analyte) on its own is not necessarily conclusive – what if the sample was very homogeneous to begin with?

To provide contrast with sub-optimal sampling practice, a comparable Replication Experiment was also conducted on the 'as is' field sample IMS-393 (also 500g), but this time 8 pucks were produced by spooning 8 x ~60g grab samples from the bag (with visible "Brazil nut effect", surely biasing the later scoops to have higher fines content). This scenario (scooping sample from a bag) is not uncommon in the rough-and-tumble field setting where time is money.

For the WA Goldfields sample an alternate "poor subsampling" method was also used for comparison, in which four specimen rocks were selected at random from a second 500g sample.

These required initial fragmentation with a hammer, before crushing, milling, and pressing duplicate pucks for each. One of the pucks failed to press (due to the high quartz content and lack of binder), so only 7 results were recorded, though enough to still allow a comparison Replication Experiment.

Credit: Block10; used with permission.

Figure 18: WA Goldfields sample specimens – pre fragmentation

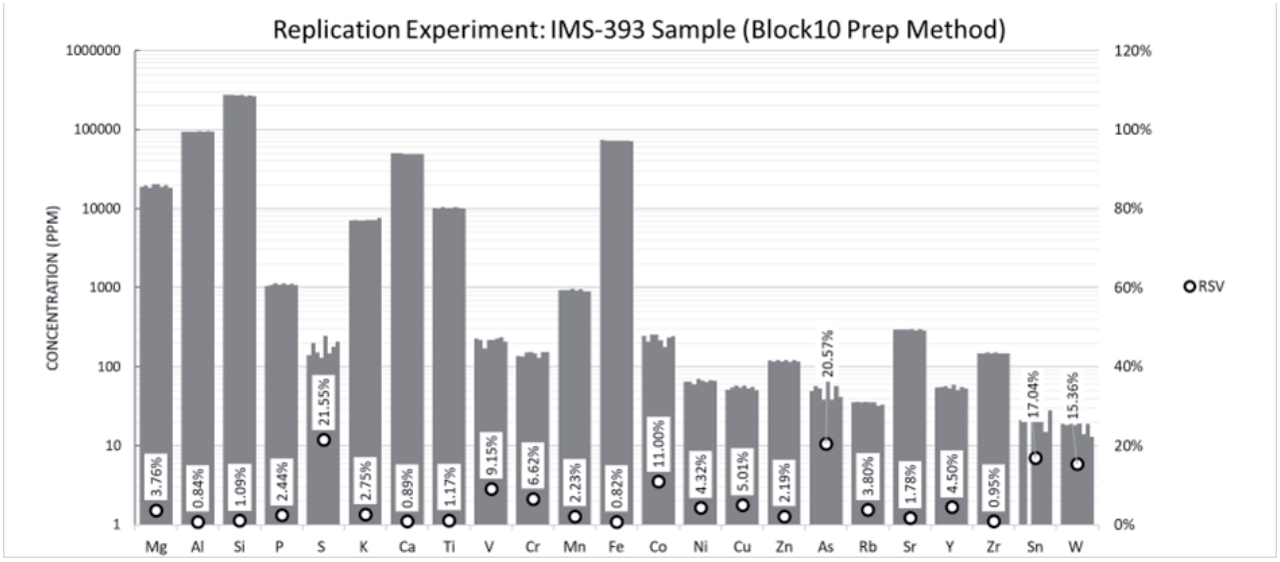
Credit: Block10; used with permission.

Figure 19: WA Goldfields specimens – post primary fragmentation

The results were compelling across the multielement suite of 23 analytes (other elements, at or near the limits of detection, were not included). For the well- prepared IMS-393 (plotted in Fig. 20 as 8-way bar clusters for each element analysed) the only elevated variability occurs in several minor or trace elements, and even then it is relatively consistent.

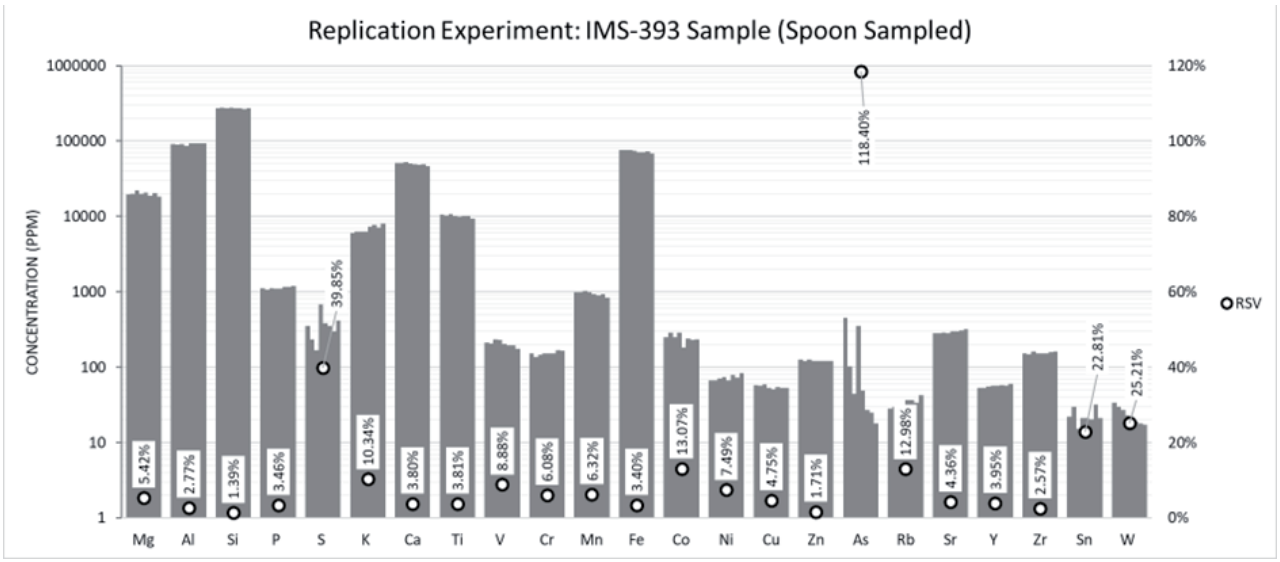
The spooned sample (grab sampling) (Fig. 21) shows visibly higher variation across most elements, and a calculation of the improvement ratios (see Tab. 4) show a significant improvement in RSV across all major (Mg Al Si P K Ca Ti Fe) and most minor/trace elements (S Mn Sr Zr Ni Sn W As Rb), with V, Cr, Co, Zn, Cu and Y showing no improvement.

Consider that although the CrushedCRM™ is intended to feature some heterogeneity, it is nonetheless a standardised product, so it is significant that there was such a discernable difference between poorer practice, and Block10 sub-sampling methodology.



Credit: Block10; used with permission.

Figure 20: Replication Experiment results: well prepared IMS-393 sample (Heavy circles: RSV%).

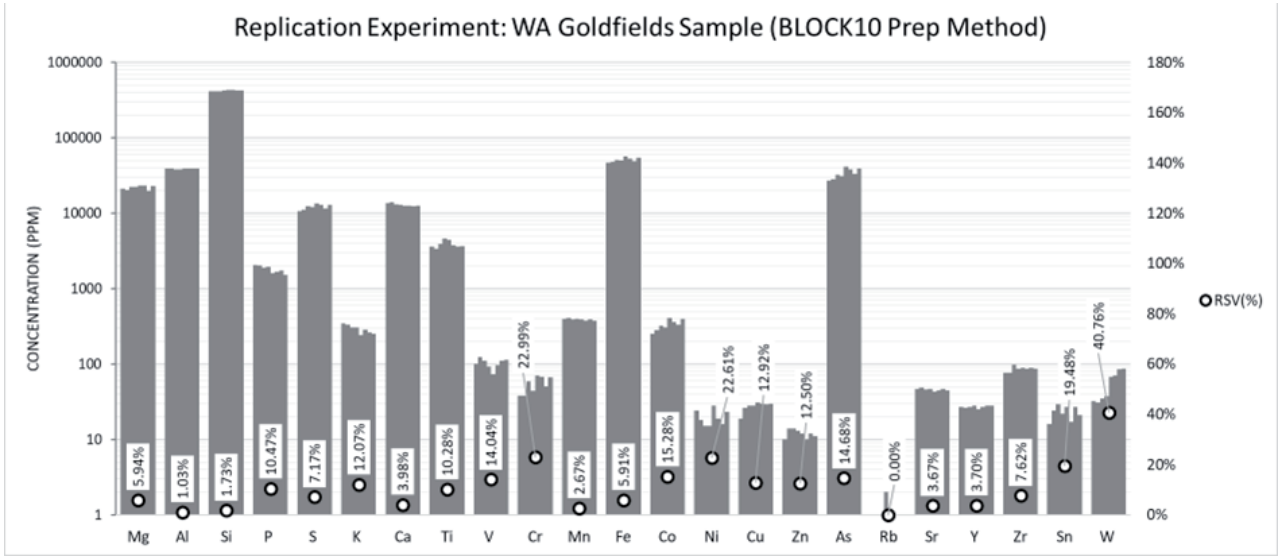


Credit: Block10; used with permission.

Figure 21: Replication Experiment results: spooned IMS-393 sample (Heavy circles: RSV%).

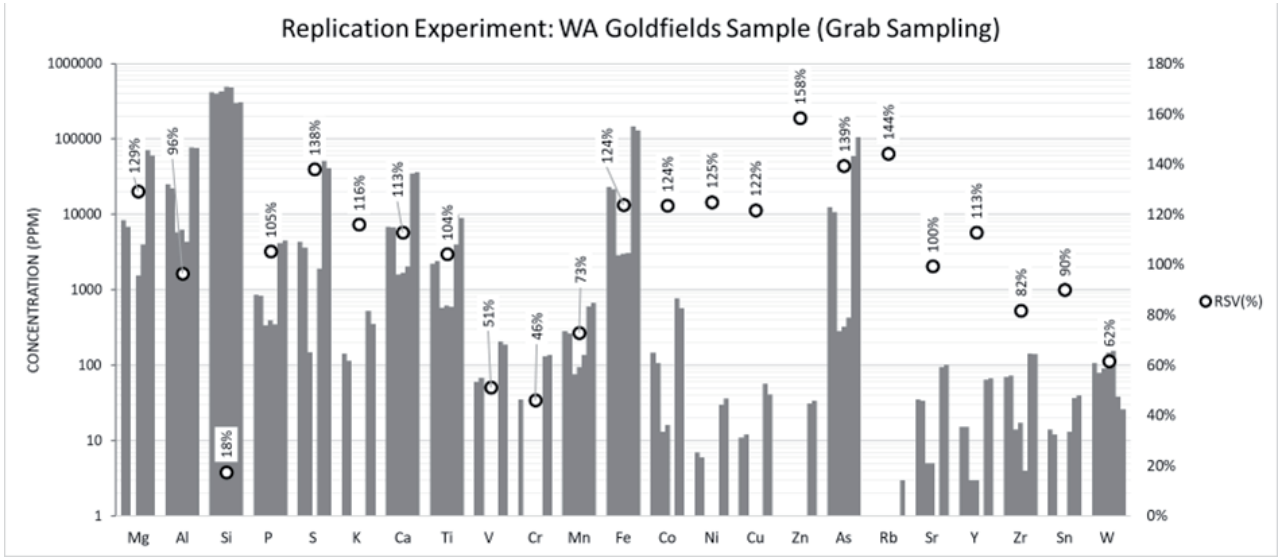
Table 4: IMS-393 Replication Experiment – analyte improvement ratios between well prepared and spooned samples.

Impact	Impact Ratio	Major Elements (>1%)	Minor Elements (0.1% - 1%)	Trace Elements (<100ppm)
No Impact	0.8x – 1.2x		V Cr Co Zn	Cu Y
Improvement	1.2x – 3x	Mg Si P	S Mn Sr Zr	Ni Sn W
Major Improvement	3x – 6x	Al K Ca Ti Fe		As Rb



Credit: Block10; used with permission.

Figure 22: Replication Experiment results: well prepared WA Goldfields sample (Heavy circles: RSV%).



Credit: Block10; used with permission.

Figure 23: Replication Experiment results: grab sampled WA Goldfields sample (Heavy circles: RSV%).

The WA Goldfields sample is visibly a much more heterogeneous sample (with distinct differences between the quartz and pyritic matrices), and there are clearly visible differences between the 4 specimen rocks. The well-prepared sample (Fig. 22) showed similar traits to the CrushedCRM™ when prepared with the same Block10 methodology – many results between 1% - 5% RSV, several in the 5-15% range, and only a handful of trace elements (Cr, Ni, Sn, W) with elevated variability.

By comparison, the grabbed specimens (Fig. 23) can best be described as wildly fluctuating – with 100% RSV across many elements, and a distinct lack of concentration across many elements for the grab samples with visibly higher quartz (lines 3,4,5 in each 7 results cluster). This sample provides an even clearer contrast and strengthens the conclusion that the Block10 equipment, following the correct field sample preparation methodology, produces highly consistent results even for a very challenging rock type.

14. Conclusions

The Block10 Sample Preparation Methodology is presented for the first time in this document, with Replication Experiments and other comparative examples across gold, copper and iron ore samples. This inaugural performance evaluation, although based on a limited experimental layout, demonstrates encouragingly accurate, high-precision results for field sample preparation for multielement pXRF analysis. Concerns over the latest sub-sampling stages using a grab sampling scoop as the final aliquoting tool have been addressed. As a quality assurance evaluation, Replicate Experiments specifically covering the procedural steps after the coarse comminution process (the jaw crusher) were performed. This means that measurement uncertainty (MU) contributions from preceding steps in the full 'lot-to-aliquot-to-analysis' pathway are not included in the present results, Tab. 4 (also see Further Work).

A coefficient of variation in the low single digits (<3% RSV) is achieved when using the BX-C jaw crusher, BX-M disc mill, BX-P hydraulic puck press, BX-R riffle splitter and BX-D dust extraction equipment to produce high quality pucks.
The BX-R riffle splitter was subjected to an augmented test regimen across a wide range of sample concentrations using a proxy rice & chia seeds mixture, which indicates a bias of less than 1% and a precision within 3% RSV at concentrations above 1%, and within 1% RSV at most higher concentrations above 10%.

15. Further Work

The full sub-sampling pathway, and further riffle splitter tests, shall be conducted to include comparison with other techniques and equipment, across a broad range of concentrations, repeated with different rock types and varying particle types, - size and density. A comprehensive DOE (Design of Experiments) approach shall be invoked.

Further test-work is also needed to quantify contamination levels for all wear part materials (tungsten, hardened steel, etc.), to guide wear part materials selection for specific sample types. With regard to carry-over and cross contamination, staggered blank / high grade tests will help quantify the impact, and validate the necessary cleaning regimes for optimal performance.

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References

BLOCK 10. (1911, June 15). Barrier Miner (Broken Hill, NSW: 1888 - 1954), p.2. Retrieved September 20, 2024, from http://nla.gov.au/nla.news-article45155491

BROKEN HILL BLOCK 10 MINE. (1911, July 22). The Sydney Morning Herald (NSW : 1842 - 1954), p. 15. Retrieved September 20, 2024, from http://nla.gov.au/nla.news-article28137517

Esbensen, K. H., & Wagner, C. (2017). Representative mass reduction in the laboratory: riffle splitting galore (with or without errors). Spectroscopy Sampling Column, 29(1).

Harris, W. J. (1909). Photo - Block 10 Mine. Retrieved September 20, 2024, from https://archivesonline.uow.edu.au/nodes/view/699

IMDEX. (2024, April 17). News. Retrieved September 20, 2024, from https://www.imdex.com/news-knowledge/news/block-10-to-be-new-vendor-of-reflex%E2%84%A2-crusher,-mill-and-press

Khan, A. (1968). Critical evaluation of powder sampling procedures. Master's thesis, University of Bradford, West Yorkshire, United Kingdom.

MicroXRF. (2024). Retrieved September 20, 2024, from https://www.microxrf.com.au/xrf-vs-micro-xrf

Nenuwa, O. B., Oke, O. O., & Sanya, O. T. (2018). Journal of Advanced Research in Manufacturing, Material Science & Metallurgical Engineering, 5(1&2), Pp 15-21.

Portable PPB. (2024, September 20). Retrieved from https://portableppb.com/our-technology/

Portable Spectral Services. (2024). Tips & Tricks for the collection of quality portable XRF data. Retrieved September 20, 2024, from https://www.portaspecs.com/tips-tricks-for-the-collection-of-quality-portable-xrf-data/

Rohiman, A., & Arifin, A. (2020). Comparation of Pressed Powder Pellet and Fused Glass Bead Preparation Techniques for Mayor Elements Analysis of Rock Samples using X-Ray Fluorescence (XRF).

XRF Scientific. (2024). XRFuse 6. Retrieved September 20, 2024, from https://www.xrfscientific.com/products/xrfuse-6/