

Achieving homogeneity in iron ore chip CRMs using a high frequency rotary splitter

By Craig Hamlyn, 18th November, 2016

This report documents the capability of ORE Pty Ltd's new high frequency rotary splitter to produce fit for purpose homogenous chip certified reference materials (CRM's) from heterogeneous source materials. It has a capacity to process batches in excess of 10 tonne and operates at 280 doses per minute.

A well-known challenge in producing homogeneous splits from a crushed iron ore sample is the prevention of particle size segregation during the subsampling (splitting) process. This requirement exists because of the contrasting comminution properties of the constituent minerals whereby aluminosilicates tend to accumulate preferentially in the fines relative to the coarser fractions. For this reason rotary splitting is generally regarded as the best method of producing representative samples, although some in the industry remain skeptical about its efficacy. The considerable advantages of chip RM's over the more commonly used pulp RM's are i) chip RM's monitor the entire laboratory process as they require an identical sample prep to field samples, such as drying, crushing, splitting and pulverization, and ii) they are indistinguishable to field samples and hence unrecognizable as RM's to the laboratory.

BACKGROUND

In 2014 ORE Pty Ltd was commissioned by a major iron ore miner to prepare two iron ore chip RM's to a nominal <10mm particle size. Each RM had a batch size of 6000kg and was rotary split via multiple steps into 2kg units (3000 units per RM). To preserve anonymity the RM's are herein referred to as CHIP CRM1 and CHIP CRM2.

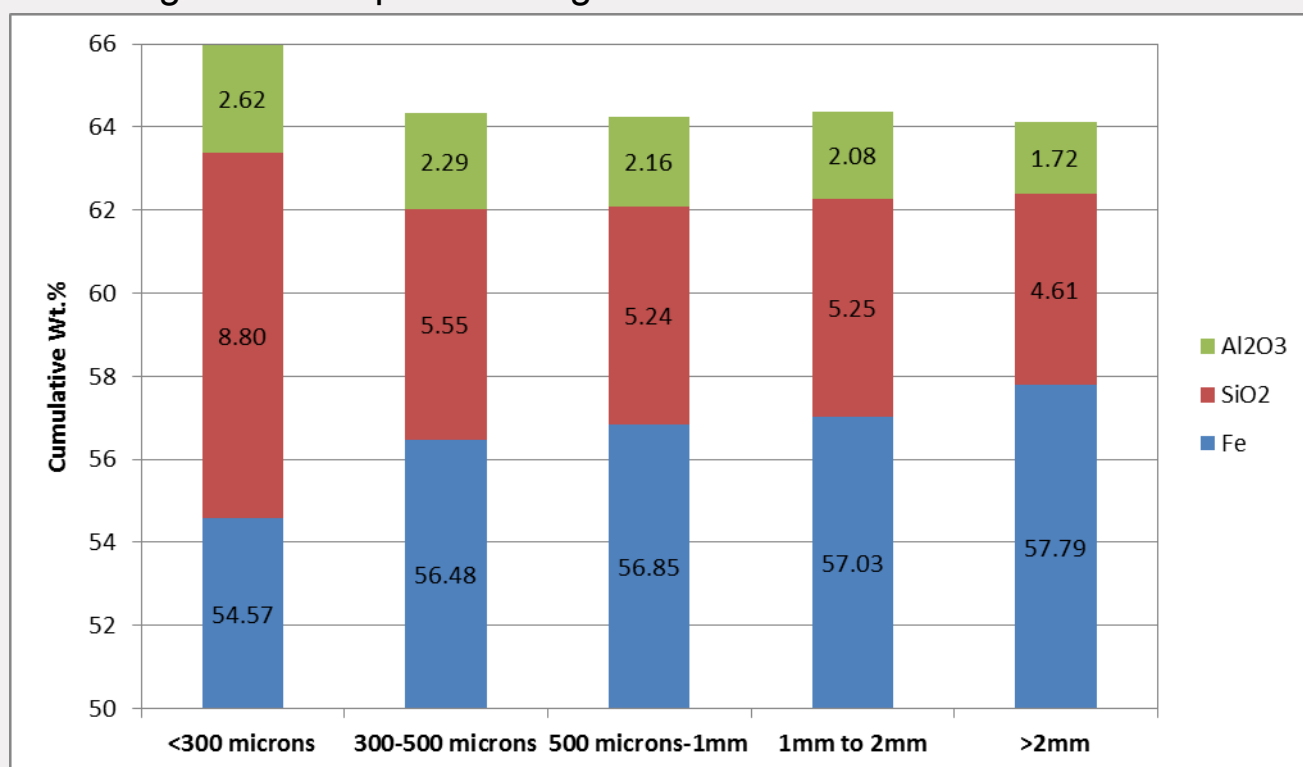
A homogeneity verification program involved the selection of twenty 2kg units selected at evenly spaced, set intervals during the packaging stage to represent the entire 6000kg batch. Each unit was completely pulverised to >95% passing 75 microns, then paired 20g subsamples were taken from each to derive 40 x 20g subsamples in total. Whole rock XRF data for these samples were tested for variability to calculate the percent relative standard deviations (RSD) within and between the 20 pairs, i.e. within and between 2kg units. The very low RSD's indicate excellent homogeneity across 2kg units of the chip CRM's. It was concluded that all 2kg chip units are essentially identical displaying a level of variance close to the analytical precision of the XRF method and comparable to that observed in high quality pulp CRMs.

HETEROGENEOUS NATURE OF SOURCE MATERIALS

The current investigation was undertaken to determine if the excellent uniformity across 2kg units of either CRM was an attribute inherited from a homogeneous source material or due to the performance of our unique high frequency rotary splitter. To evaluate heterogeneity and its particle size dependence within the source materials comprising CHIP CRM1 and CRM2 a random 2kg unit of each was selected and screened into five size fractions: <300 μm , 300-500 μm , 500 μm -1mm, 1-2mm and >2mm. The composition of each size fraction was established by XRF for basic iron ore package elements. The results for Fe, SiO₂ and Al₂O₃ showed a strong correlation between grain size and chemical composition (minor analytes, such as TiO₂, Mn, P, S, MgO and K₂O, were close to detection levels so reading resolution errors prevent their inclusion in this study).

Figures 1 and 2 below show the Al₂O₃, SiO₂ and Fe concentrations in each size fraction for CHIP CRM1 and CHIP CRM2. To maintain confidentiality an offset has been applied to the analytical results depicted in both graphs. The compositions of both CRM's can be seen to be highly heterogeneous across the various size fractions. SiO₂ and Al₂O₃ are concentrated in the fines and Fe concentrated in the coarse fractions, presumably in response to contrasting friabilities of aluminosilicates and Fe oxide minerals.

Figure 1. Composition vs grain size fraction for CHIP CRM1.



Tables 1 and 2 compare the Relative Standard Deviations (RSD's) for Fe, SiO₂ and Al₂O₃ between the five size fractions to the RSD's within and between 2kg units for CHIP CRM1 and CHIP CRM2, respectively. The within-unit RSD's are generally lower than the between-unit RSD's and are considered to approximate laboratory measurement errors for the three analytes. The marginally higher between-unit RSD's reflect a modest sampling error for the 2kg units that is dwarfed by the between size fraction RSD's shown in red in the tables.

Figure 2. Composition vs grain size division for CHIP CRM2.

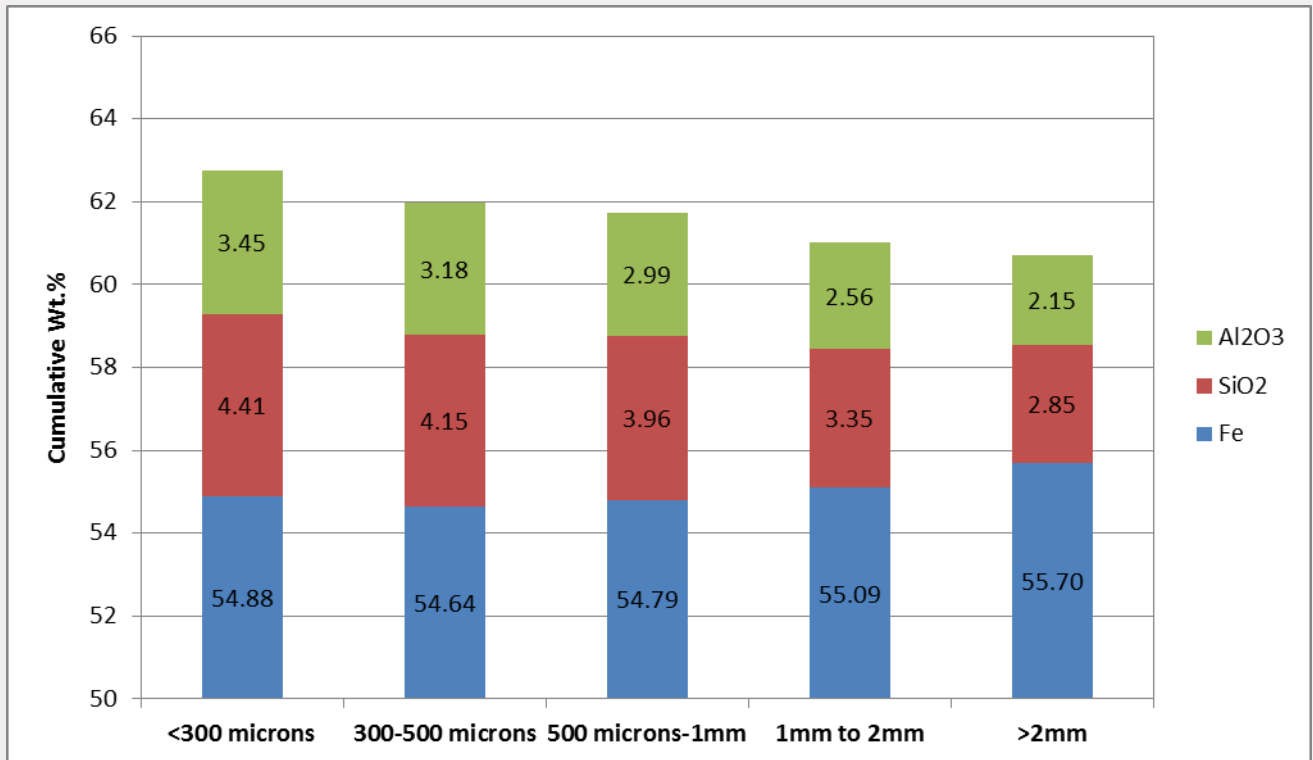


Table 1. RSD's for CHIP CRM1.

Type	Analyte	Within (RSD)	Between (RSD)
Grain Size Fractions	Fe		3.00%
Whole 2kg CRM	Fe	0.25%	0.20%
Grain Size Fractions	SiO ₂		39.86%
Whole 2kg CRM	SiO ₂	0.24%	1.04%
Grain Size Fractions	Al ₂ O ₃		21.32%
Whole 2kg CRM	Al ₂ O ₃	0.45%	0.55%

Table 2. RSD's for CHIP CRM2.

Type	Analyte	Within (RSD)	Between (RSD)
Grain Size Fractions	Fe		1.06%
Whole 2kg CRM	Fe	0.13%	0.20%
Grain Size Fractions	SiO ₂		23.97%
Whole 2kg CRM	SiO ₂	0.52%	0.95%
Grain Size Fractions	Al ₂ O ₃		25.46%
Whole 2kg CRM	Al ₂ O ₃	0.51%	0.91%

The contrasting RSD's are graphically depicted in Figures 3 and 4. RSD's are plotted for between the five grain size fractions (see square prisms in figures below) together with the RSD's within and between the twenty 2kg CRM units prepared by rotary splitting (see cylindrical prisms in figures below). The 'grain size fraction' RSD's were calculated from the means of duplicate determinations of the five grain size fractions following pulverisation. The

within and between 2kg unit RSD's were calculated from duplicate determinations from twenty 2kg rotary split samples. Samples for analysis were obtained by conventional laboratory preparation of each 2kg chip unit (crush to -2mm, pulverise whole sample to 95% -75µm, homogenise and subsample for XRF analysis). Figures 3 and 4 clearly show elevated heterogeneity between grain size fractions (red square prisms) compared to the within and between whole 2kg CRM sample variances.

Figure 3. Comparison of RSD's for CHIP CRM1.

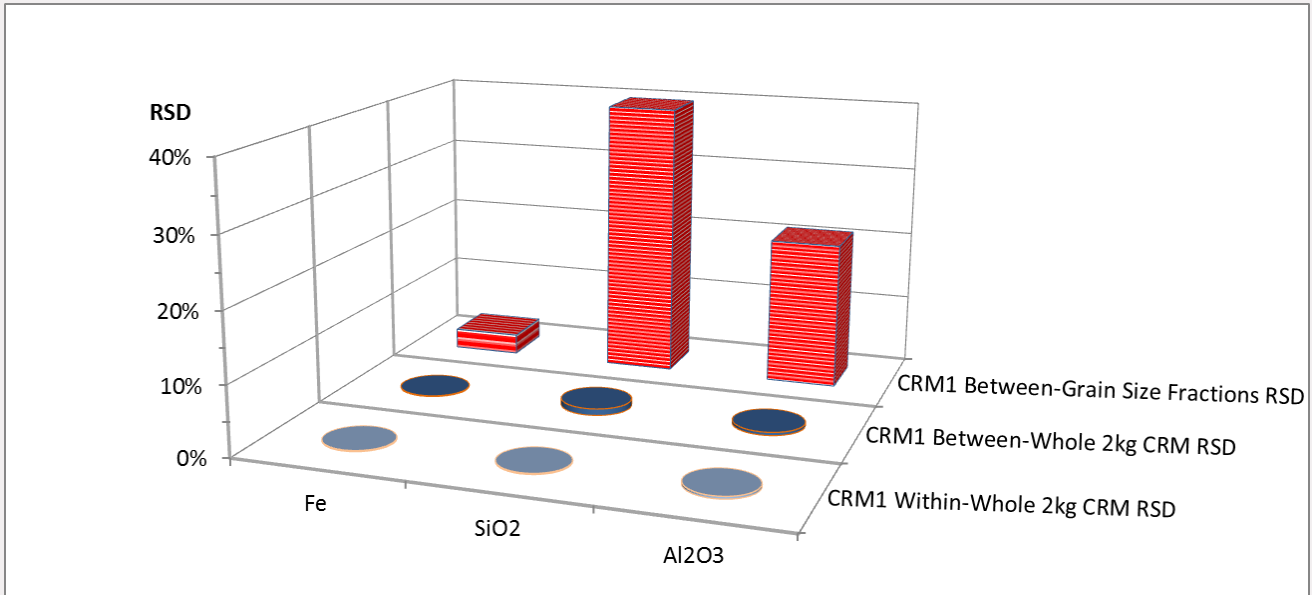
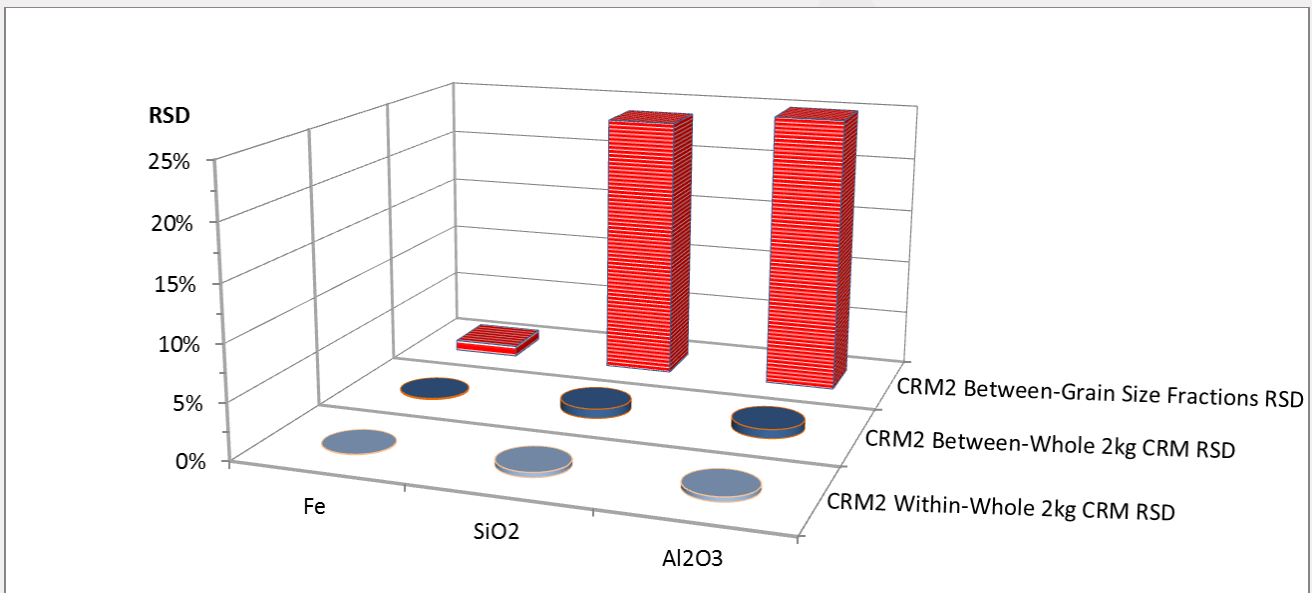


Figure 4. Comparison of RSD's for CHIP CRM2.



This compositional variance due to grain size effects and segregation is successfully eliminated by the high frequency rotary splitter. The very low RSD's for the between 2kg CRM samples are comparable with those seen in pulp CRM's. Both CRM's exhibit a high degree of compositional homogeneity across 2kg units that is close to the analytical precision of the

fusion XRF method and are considered fit for purpose as routine grade control chip CRM's. Unlike pulp CRM's, which can only monitor the analysis step, chip CRM's provide control over the entire process from sample preparation through to analysis. They are also indistinguishable from field samples and hence blind to laboratory personnel.

The results of this study demonstrate the ability of ORE's high frequency splitter to produce compositionally identical 2kg chip CRM's from heterogeneous materials. Subsequent to this trial, five additional chip CRM projects have been undertaken with other major mining houses and have yielded the same level of success.

REFERENCES

ISO Guide 35 (2006), Reference materials - General and statistical principles for certification.