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Design advances and operational studies for the True Pipe® Sampler: A symmetry based unit for reliable sampling of pressurised particulate streams

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Obtaining representative samples with minimised sampling errors, is critical for calculating accurate metallurgical mineral balances on process plants. A challenging situation exists, where no acceptable, robust or economically viable sampler has been commercialised for sampling of one-dimensional pressurised slurry pipelines yet. The design of the True Pipe® in-line sampler is based on the principle of symmetry, as described by Dominique François-Bongarçon[7], and also operates on a fail-safe principle for control on the synchronous opening and closing of identical valves for the sample chamber. Previous test work on the True Pipe® in-line sampler indicated that the prototype sampler is reliable within certain tolerances, initially indicating the concept could well be a viable design option. This paper presents the results from further test work, which mainly investigated three sampling phenomena in more detail, by examining classical one dimensional sampling, with the aid of an automated valve actuator. Firstly, the transient effect, which originates from the disruption in laminar particle flow. Secondly, the effect of split sampling, where the portion of the stream is sampled, as well as the full stream. Thirdly, the effect of symmetry is confirmed. The expected accuracy level of the True Pipe® in-line sampler is also evaluated for varied material conditions. Advances on the design include the ability to sample the entire pressurised particulate stream in a safe operating condition, by making use of a mechanical actuator for synchronous opening and closing of the sample chambers, as well as improved control on the valve opening and closing cycles.

Introduction

The need for increased accuracy of sampling in a mining process plant environment is driven by the reliability and confidence level that can be placed on the samples used for metallurgical accounting, official company reports and financial statements to comply with the increased requirements of corporate governance principles and guidelines as laid out in the AMIRA code[1]. Case studies[2] show that incorrect sampling protocols and equipment may have a crippling financial effect for the mine, but applying correct theory of sampling may save money. These studies underline the importance of installing correct sampling equipment on tailings streams.

Tailings streams are often pressurised horizontal one-dimensional pipe lines, which limit the application of conventional cross stream sampling equipment and for which there has to date not been a robust reliable TOS-compliant sampler solution presented. Some options available for sampling pressurised slurry streams are the t-piece bypass valve, poppet samplers and pressure pipe samplers. All of which fail to comply with TOS principals[3].

To honour the fundamental rule for correct sampling and sample processing, all parts of the ore, concentrate or slurry to be sampled must have an equal probability of being collected and becoming part of the final sample for analysis[4,5]. Poor precision may be improved by replicate samples or stringent control on sample preparation and analysis[6], but this will not eliminate bias once it is present. Correct design of sampling equipment and sampling systems can help to eliminate or at least minimise sources of bias to acceptably low levels.

Upholding the principle of symmetry[7], proposed by Dominique François-Bongarçon, where any biasing mechanism should affect the sample and its reject in exactly symmetrical ways, ensures sample correctness.

Design of the True Pipe® in-line sampler

The designed application for the True Pipe® in-line sampler is for sampling of high pressure particulate streams in the mining process plant environment, currently specifically focussing tailings pipe lines where flow velocity of 6ms^{-1} and line pressure of 1600 kPa or more is common. The True Pipe® in-line sampler, shown in Figure 1, allows for two parallel flow paths with diameters equal to that of the main feed pipe to the device, which is connected with a small angle Y-piece at each end. Two valves are present in each of the flow paths, delimitating specific sample captured volumes.

Previous True Pipe® in-line sampler test work

Initial exploratory test work conducted on the True Pipe® in-line sampler last year showed that at a 95% confidence interval, no statistically significant difference could be detected on the difference in mean between the reference sample and sample from each of the two sampler legs, when a synthesized ferrosilicon-silica ore was tested in the unit[3].

Drawbacks on the initial design included:

- Difficult manual operation of valves
- Insufficient control on valve closing time
- Unsafe operational condition for sampling a full cross stream cut
- Synthesized ore did not fully represent fluid rheology as present in mineral processing plants
- Manual sample draining and extraction

Design improvements on True Pipe® in-line sampler

The design optimisation of the True Pipe® in-line sampler calls for evaluation of certain theories associated with fluid born particle sampling. Transient effect recognition, in order to establish the final plant footprint required for the sampler, is where the actuating of the valves would cause an upstream disturbance of the particulate

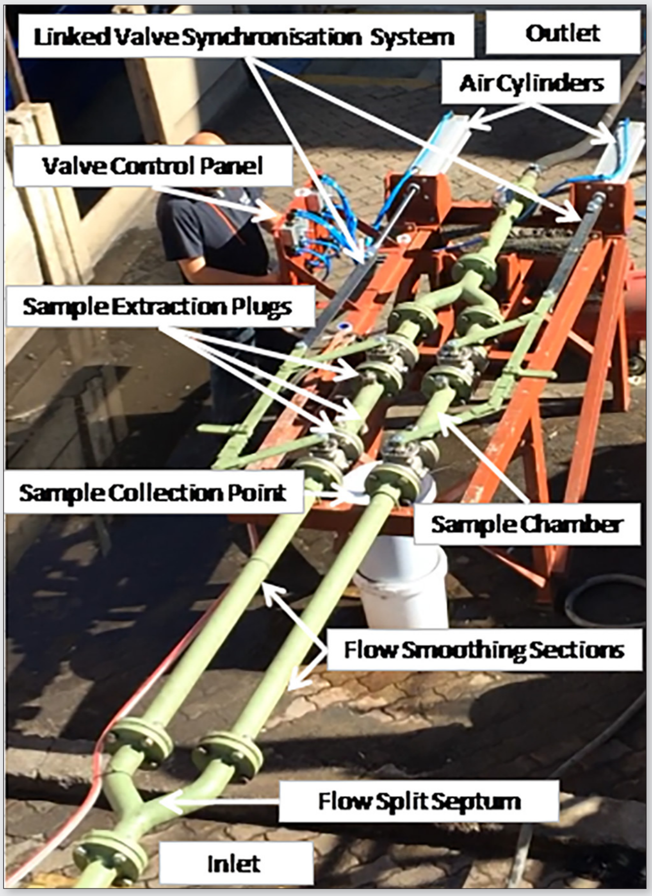

Figure 1. True Pipe® in-line sampler assembly

fluid stream at the septum of the Y-piece. Provided the valve closing time is faster than the time required for the disturbed particles to reach the first valve, the sample should be identical to the parent stream. This was to be evaluated by testing two different lengths of flow smoothing sections.

Another design consideration is identification of bias imposed on the particulate stream by split sampling. Inherent constitutional and distributional heterogeneities dismiss the theory of split sampling, where it is accepted that if a fluid stream is halved exactly, that each half of the stream is identical to the parent stream. Meticulous care was taken in the manufacturing of the device to ensure the best possible axial symmetry to halve the particulate flow.

The design of the True Pipe® in-line sampler respects the principle of symmetry by synchronous closing of actuated valves in the pipe line by means of a linked synchronising system. The principal is applied where the effect of any improperly delimitated particles misplaced into the sample chamber by the first valve would be identical to the effect of any particles improperly delimitated to the outside of the sample chamber by the second valve. One of the largest design improvements of the True Pipe® in-line sampler is the addition of actuators for automated control on consistent valve closing. The addition of the pneumatic actuators enables synchronous open and closing of either both valves in the same line (with the additional valves remaining open in the fail-safe position), or opening one set of valves and closing the other synchronously to enable cross stream sampling of the entire flow. Consistency of the valve closure speed was maintained by the addition of a pressurised air reservoir to constantly supply air to the control point where the air pressure to the valves is measured and monitored.

Methodology

A bias identification approach was followed in the design and operational assessment of the True Pipe® in-line sampler. This requires the sampler in question to be tested against a more recognised, reliable, correctly designed and unbiased sampling unit[8]. The integrity of the reference sample was ensured, by making use of an automated Multotec vezin sampler, which is TOS compliant, to obtain reference samples.

Test rig set-up

A closed re-cycle system as shown in Figure 2, consisting of a bottom discharge 5 m^3 feed tank and 6/4 AH slurry pump fitted with a variable speed drive, which was run at 850 rpm to achieve a measured in-line pressure of 300 kPa, which was the maximum pressure safely attainable with the test rig. Continuous mixing is established by aerating the feed tank with a Pachuca valve and recirculation of the particulate stream back into the feed tank. Class 10 2.5 inch industrial fibre reinforced rubber hose was used to connect the pump outlet to the sampler inlet and the sampler outlet to the vezin sampler feed chute. A special support structure, to which the sampler was secured with U-bolts, was manufactured to ensure level installation of the True Pipe® in-line sampler and to support the two additional 160 mm air cylinder valve actuators attached to the synchronization linkage system for automated valve closure.

Material handling and sampling

Chrome tailings ore with a top size of 425 µm, from a chrome mineral processing plant discard line in the South African Bushveld Igneous Complex was sourced for this test work. Additional silica sand with a top size of 1 mm was used for the test which required the ore composition to be synthetically altered. A slurry make up of approximately 45% solids by weight was maintained for all tests, except on the evaluation of this variable, where the solids were decreased to 25%.

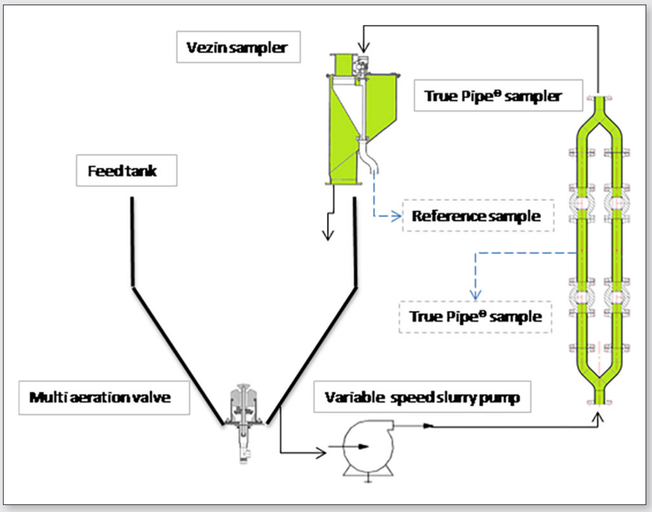

Figure 2. Schematic test rig setup for evaluating the True Pipe® in-line sampler

Samples are extracted from the sample chamber by washing particles out of the chamber and draining the sample into a container which is sealed immediately after sample extraction. Samples were dried at 90°C and the dry samples were dis-agglomerated and homogenised before being representatively split to a sample mass exceeding the prescribed minimum sample mass[3].

Representative sample splits were chemically analysed for major elements, specifically %Cr and %Si, by inductively coupled plasma atomic emission spectroscopy (ICP with OES), for which a certified reference material of similar matrix was used in the calibration of the instrument.

- For split sampling evaluation, a sample from the reference vezin sampler and each of the True Pipe® in-line sampler streams at 50% flow was taken. All other samples are collected from a full cross cut or slice of the particulate stream for each of the two legs.
- Transient effect recognition was enabled by fitting a 250 mm pipe extension before the sampling chamber and comparing this to a 1400 mm pipe extension where no transient fluid effects would be expected to be active.
- The application of the principle of symmetry was tested by introducing different valve closure speeds, all of which were synchronous and now automated.
- The operational effect of a change in solids concentration was evaluated by adding more water to the slurry make-up.
- Heterogeneity effects resulting from a change in ore type was tested by the addition of coarse silica sand to make up a synthesised ore.

Statistical methodology

The student's t-test is deemed satisfactory for obtaining 95% confidence limits on sampling data with reasonably consistent sample masses[8], when sample means or sample mean differences for two sample sets are compared. For comparison of three or more sample sets, repeated analysis of variance (rANOVA) is recommended. Both of these tests yield a two tailed p-value, which based upon a 95% confidence limit ($\alpha = 0.05$), is used to evaluate if the null hypotheses should be rejected.

Hypothesis tests[9] were conducted to evaluate the effect of design and operational variables on the %Cr mean difference between the reference vezin sample and the True Pipe® sample pair, (H_0: $\mu_{Reference} - \mu_{Sampler} = 0$; H_a: $\mu_{Reference} - \mu_{Sampler} \neq 0$). This test indicates whether the mean of the reference sample and the mean of the True Pipe® sample are statistically different.

An additional hypothesis test was conducted to evaluate whether the difference in sample mean between the reference vezin sample and the True Pipe® sample pair were statistically different under different design and operational conditions, (H_0: $[\mu_{ReferenceA} - \mu_{SamplerA}] - [\mu_{ReferenceB} - \mu_{SamplerB}] = 0$; H_a: $[\mu_{ReferenceA} - \mu_{SamplerA}] - [\mu_{ReferenceB} - \mu_{SamplerB}] \neq 0$). When the null hypothesis is accepted, the difference in mean between the reference and its corresponding True Pipe® sampler sample is not statistically different from the difference between another reference sample and its corresponding True Pipe® sampler sample. Thus accepting the level of uncertainty was not affected by the change in design, or operational variable for that test.

The use of rANOVA to test the equality of sample means is kept to analysis of split flow sampling means, where a vezin reference sample is compared to two samples from the True Pipe® sampler, each representing a 50% split of the original particulate stream. When a statistically significant difference exists between at least one of the True Pipe® sampler sample means and the vezin reference sample mean, the rANOVA hypothesis test should reveal this, (H_0: $\mu_{Reference} = \mu_{SamplerA} = \mu_{SamplerB}$; H_{a1}: $\mu_{Reference} \neq \mu_{SamplerA} = \mu_{SamplerB}$; H_{a2}: $\mu_{Reference} = \mu_{SamplerA} \neq \mu_{SamplerB}$; H_{a3}: $\mu_{Reference} \neq \mu_{SamplerA} \neq \mu_{SamplerB}$), although it is unable to identify which of the alternative hypothesis are valid.

Results and discussion

Flow 50% split sampling

The results from the split sampling rANOVA evaluation in Table 1 indicate that there exists a statistically significant difference between the sample means of the vezin sampler, True Pipe® Right sample and True Pipe® Left sample. The F-value of 9.05 exceeding the critical F-value of 3.19 and p-value of 0.00004, leads one to reject the null hypothesis of equality on sample means. The p-value of 0.044 on the paired t-test in Table 2, evaluating the mean difference in %Cr content between the vezin sample and the corresponding side

Table 1. Hypothesis rANOVA test on flow 50% stream splitting

Source of Variation	SS	df	MS	F	P-value	H_0: $\mu_{Reference} = \mu_{SamplerA} = \mu_{SamplerB}$
Between	0.51	2	0.26	9.05	0.00004	Reject Null Hypothesis
Within	3.97	72				
Subjects	2.61	24				
Error	1.36	48	0.03			

($\alpha = 0.05$, SS = sum of squares, df = degrees of freedom, MS = mean squares, F = F-statistic)

Table 2. Hypothesis paired t-test on flow 50% stream splitting

	Sample Size (N)	Mean %Cr (μ)	Sample Variance (σ)	Mean Difference ($\mu_{Reference} - \mu_{Sampler}$)	Variance (s^*)	P-value
Reference	25	11.69	0.061			
True Pipe® Right	25	11.62	0.047	0.073	0.043	0.044
True Pipe® Left	25	11.49	0.056	0.200	0.050	
H_0: $[\mu_{ReferenceA} - \mu_{SamplerA}]$- $[\mu_{ReferenceB} - \mu_{SamplerB}] = 0$				Reject Null Hypothesis		

($\alpha = 0.05$)

Table 3. Hypothesis t-tests on transient vs flow smoothing effect recognition

	Sample Size (N)	Mean %Cr (μ)	Sample Variance (σ)	P-value	Mean Difference ($\mu_{Reference} - \mu_{Sampler}$)	Variance (s^*)	P-value
Reference A	19	12.19	0.016	0.00000	0.517	0.033	0.013
True Pipe® Transient	19	11.67	0.014				
H$_0$: $\mu_{Reference} - \mu_{Sampler} = 0$		Reject Null Hypothesis					
Reference B	18	11.60	0.024	0.00001	0.341	0.050	
True Pipe® Non-Transient	18	11.94	0.022				
H$_0$: $\mu_{Reference} - \mu_{Sampler} = 0$		Reject Null Hypothesis					
H$_0$: $[\mu_{ReferenceA} - \mu_{SamplerA}] - [\mu_{ReferenceB} - \mu_{SamplerB}] = 0$					Reject Null Hypothesis		

($\alpha = 0.05$)

of the True Pipe® sample, indicates only a marginally statistically significant difference between the bias in True Pipe® Right and True Pipe® Left samples.

This indicates that the extreme effort to axially symmetrically split the particulate stream in two 50% flow streams does not help to eliminate a significant bias between the reference sample mean and the individual True Pipe® samples, but that this bias could be equal on each side of the True Pipe® sampler. This is a significant detail that should not be missed when one wishes to employ partial sampling techniques of plant process streams during process control operations.

Transient effect recognition

It is clear from both hypotheses tests in Table 3 that not only are there significant differences between the samples exposed to the transient disturbance and the samples which were subjected to a flow smoothing section prior to sampling with respect to their individual reference vezin samples, but also that there exists a statistically significant difference in the bias generated from each scenario. A p-value of 0.013 on the mean difference between the two design types shows that the non-transient True Pipe® sample, where a flow smoothing pipe of 1650mm was added before the sampling chamber, has a smaller bias than the transient True Pipe® sample, where there was no intentional flow smoothing.

Principle of symmetry

By throttling the main air valve to the system, the speed of automated valve closure was reduced consistently by a constant reduction in the air supply to the valve actuators from 8 bar to 4 bar. Table 4 indicates that again the means of the True Pipe® samples were not equivalent to the means of their paired reference vezin samples, but that the mean difference between the True Pipe® samples and vezin reference samples was not statistically significant, where a p-value of 0.441 does not reject the hypothesis, that the difference in sample means are equivalent.

These results validate the principle of symmetry, where the bias imposed by a disturbance in the particulate stream will be countered symmetrically if an identical disturbance is introduced at either end of the delineated sample.

Stream composition effect

A change in the particulate stream make-up, where the solids by weight concentration was changed from 45% to 25% did not improve the equality of True Pipe® sample means and vezin reference sample means in the 95% confidence limit hypothesis test results shown in Table 5, but with a p-value of 0.529 on the mean difference evaluation between the two True Pipe® sample sets, the bias in these two scenarios appeared to not be statistically significantly different. Thus the change in solids content of the fluid stream, did not affect the precision of the sampler.

Ore composition effect

The addition of silica sand with a top size of 1mm to the standard chrome tailings ore sourced for this test work highlighted the heterogeneity effect in sampling in this test. By measuring a statistically

Table 4. Hypothesis t-tests on principle of symmetry

	Sample Size (N)	Mean %Cr (μ)	Sample Variance (σ)	P-value	Mean Difference ($\mu_{Reference} - \mu_{Sampler}$)	Variance (s^*)	P-value
Reference A	18	11.94	0.024	0.00001	0.341	0.050	0.441
True Pipe® Slow Valves	18	11.60	0.022				
H$_0$: $\mu_{Reference} - \mu_{Sampler} = 0$		Reject Null Hypothesis					
Reference B	19	10.79	0.049	0.00000	0.399	0.050	
True Pipe® Fast Valves	19	10.39	0.018				
H$_0$: $\mu_{Reference} - \mu_{Sampler} = 0$		Reject Null Hypothesis					
H$_0$: $[\mu_{ReferenceA} - \mu_{SamplerA}] - [\mu_{ReferenceB} - \mu_{SamplerB}] = 0$					Do Not Reject Null Hypothesis		

($\alpha = 0.05$)

Table 5. Hypothesis t-tests on stream composition effects

	Sample Size (N)	Mean %Cr (μ)	Sample Variance (σ)	P-value	Mean Difference $(\mu_{Reference} - \mu_{Sampler})$	Variance (s^*)	P-value
Reference A	9	12.31	0.017	0.00000	0.545	0.011	0.529
True Pipe® 45% solids	9	11.77	0.012				
H_0: $\mu_{Reference} - \mu_{Sampler} = 0$		Reject Null Hypothesis					
Reference B	9	13.18	0.037	0.00001	0.607	0.070	
True Pipe® 25% solids	9	12.57	0.099				
H_0: $\mu_{Reference} - \mu_{Sampler} = 0$		Reject Null Hypothesis					
H_0: $[\mu_{ReferenceA} - \mu_{SamplerA}] - [\mu_{ReferenceB} - \mu_{SamplerB}] = 0$					Do Not Reject Null Hypothesis		

($\alpha = 0.05$)

insignificant difference in the means of the True Pipe® Synthetic ore sample and its corresponding reference vezin sample a higher level of accuracy is attained with the True Pipe® sampler when this change in ore composition is made. A p-value of 0.986 for the synthesized ore means in Table 6 indicates that the means of the True Pipe® sample and vezin reference sample are not statistically different. The p-value of 0.003 rejecting the equality of the difference in sample means for the original standard and later synthetic ore, shows that the performance of the sampling unit will be effected by the ore type sampled.

The statistically significant difference in bias between the two different ores may be attributed to fluid rheology effects, where the fractional contribution of clay type minerals in the ore can easily increase the viscosity of the stream. When the viscosity of the particulate fluid is decreased, the effect of particle-particle interactions on the accuracy of sampling may also be decreased. The addition of silica to the original ore decreases the particulate fluid viscosity by surface chemical interactions between the silica particles and clay minerals. Positively charged ions from the wettened clay minerals will chemically bond to the negatively charged sites on silica particles, thus decreasing the concentration of dissolved ions in the fluid. Although this in turn will decrease the particle stability in the fluid, where coarser particles tend to settle faster, maintaining a particle line velocity of 4.5 ms^{-1} overcomes particle settling in the pipe column.

Conclusion

The design of the True Pipe® in-line pressurised particulate stream sampler, based on Dominique François-Bongarçon's principle of symmetry, strives to not only minimise sampler bias, but also achieve repeatable sampling results. The principle of symmetry inherently accepts a specific level of uncertainty, which is introduced in a symmetric fashion on either end of the delineated sample, such that a nett zero effect may be obtained. This design called for evaluation of certain theories associated with fluid born particle sampling to optimise future scale up of the prototype unit for industrial application. This test work confirms that the implementation of split sampling does not yield reliable sampling results from 50 percent cross stream cuts, no matter how careful the design tolerances and reliance on trying to control the particle lines of flow. One must rather design the unit to accommodate a full cross stream cut for meaningful results which once again validates and underpins TOS. It also identified that a difference in the magnitude of bias imposed by changing the pipe configuration for transient and flow smoothed, non-transient fluid conditions. The test work showed that a smaller bias was obtained when the particulate stream was subjected to a flow smoothing section before symmetric sampling. The principle of symmetry was confirmed by results showing no statistically significant difference in the magnitude of bias, when different synchronised valve closure speeds were implemented. The results of this test work also show that operational changes, such

Table 6. Hypothesis t-tests on ore composition effects

	Sample Size (N)	Mean %Cr (μ)	Sample Variance (σ)	P-value	Mean Difference $(\mu_{Reference} - \mu_{Sampler})$	Variance (s^*)	P-value
Reference A	9	12.31	0.017	0.00000	0.545	0.011	0.003
True Pipe® Standard Ore	9	11.77	0.012				
H_0: $\mu_{Reference} - \mu_{Sampler} = 0$		Reject Null Hypothesis					
Reference B	9	8.97	0.036	0.986	-0.002	0.159	
True Pipe® Synthetic Ore	9	8.97	0.148				
H_0: $\mu_{Reference} - \mu_{Sampler} = 0$		Do Not Reject Null Hypothesis					
H_0: $[\mu_{ReferenceA} - \mu_{SamplerA}] - [\mu_{ReferenceB} - \mu_{SamplerB}] = 0$					Reject Null Hypothesis		

($\alpha = 0.05$)

as changes to the solids content of the particulate stream do not have a statistically significant effect on the sampling bias. Changes in the ore composition show a significant difference in the level of uncertainty. This phenomenon is attributed to the particle-particle interactions associated with the viscosity changes in the fluid.

The True Pipe® in-line sampler design investigations conducted to date, has sufficiently proven the concept of use for this patented sampler type as well as recognising certain effects to consider in the scale-up design. The next step is to manufacture a scale-up design of the True Pipe® sampler and also include an automated washing system, which will minimise operator interference during sample extraction.

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