MEASUREMENT OF SMELTER-GRADE ALUMINA PROPERTIES

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ABSTRACT

In assessing the quality of an alumina, customer and supplier must have confidence in the analytical methods used. A suite of smelter-grade alumina samples were analysed by both Pechiney and Alcoa of Australia using their standard methods. The properties selected for measurement were those that are considered important for aluminium smelting. The aluminas had a wide range of properties, and data for repeatability of the measurements were obtained. Excellent agreement was obtained for the chemical analyses data for sodium, iron, silicon, titanium and calcium even though different wet methods of chemical analysis were used. For phase analysis there was good agreement for alpha alumina values but for gibbsite, at the low levels of interest, large differences were obtained. Excellent agreements for sizing (both dry screen and laser diffraction), surface area, loose bulk density and L.O.I. were obtained. Differences were obtained for packed bulk density and attrition index because of differences in equipment and/or methods used. The comparison work highlighted the need for international reference standards and recognised international standard methods for properties such as attrition and bulk density. The size distribution data of sub-screen size material, which can now be reliably obtained using laser diffraction, could provide extra useful information on smelter-grade alumina, especially such sizing data for the attrition test.

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1.0 INTRODUCTION

The continual evolution of the smelting process necessitates changes in the alumina property requirements to maximize smelter efficiency. The properties of the alumina produced from the Bayer process are similarly affected by the continual process improvements to maximize the efficiency of that process, and by raw material changes such as the type of bauxite. Various chemical and physical properties of the alumina are determined and used as both alumina quality criteria and for seeking correlations with smelting performance in order to further optimize efficiency. It is vital that such properties are unambiguously defined (both between customer and supplier and among suppliers) and that the values obtained via the analytical methods are consistent. This may appear to be straight forward for chemical analyses, but would be less so for physical and empirical measurements such as sizing, surface area, dustiness etc.

Pechiney and Alcoa of Australia decided to do a direct comparison of the methods they use routinely for measuring alumina properties. As a first step, the analytical methods used to measure the basic chemical and physical properties of the alumina were compared. Understanding and quantifying any differences would assist when discussing alumina trends and effects on smelting behaviour.

The need for standard methods for measurement of smelter grade alumina has been well recognised in the past and a Committee MN/9 under the auspices of Standards Australia was formed which included all the alumina and aluminium producers in Australia. That Committee has produced several standard methods, some of which were developed by the Committee. The present comparison involves some of those methods as well as others still being assessed by the Committee.

2.0 PROPERTIES AND METHODOLOGY

The properties selected for comparative analysis were those considered the most critical in smelter-grade alumina related to metal purity and pot and scrubber efficiency. Those properties have been well documented, e.g. Homsi, (1989). Of those properties the following were selected for testing:

- Chemical : Soda, iron, silica, calcia, titania
- Phase : Alpha, gibbsite
- Physical : Loose and packed bulk densities, loss on ignition (300-1000° C), surface area, sizing by dry screens, wet screening for fines, laser sizing and attrition index

Laser sizing, although not a standard method, was included as the improved resolution and reproducibility of laser sizing equipment has the potential to both simplify sizing and give extra information especially on the nature of the fines in alumina. Laser

sizing can also be utilised in analyses such as attrition index rather than conventional screen sizing.

All samples used in the testwork were from the three Western Australian Refineries of Alcoa of Australia Limited. Some samples were taken from non-standard sampling locations and at unusual process conditions so that a wide range of values of the properties was obtained; such samples would better highlight any differences in the analyses. A total of eight samples comprising approximately 5kg of each were obtained. These samples were riffle split into four sub-samples. One sample was sent to Pechiney for analysis and one to Alcoa's Kwinana product alumina analysis laboratory. All analyses were undertaken in the normal laboratories for such alumina analyses, and by those people who routinely do the analyses, i.e. the normal level of repeatability was sought, not the best that could be achieved. Three of the eight samples were analysed in duplicate, once per week, for six weeks so as to estimate the repeatability of each analysis. The other five samples were analysed in duplicate.

3.0 CHEMICAL ANALYSES

Pechiney fuses 2.0g of alumina with 5.0g of potassium carbonate, 1.5g of lithium carbonate and 2.5g of boric acid. The molten product is dissolved in water with 10ml of concentrated sulphuric acid. The resulting solution is analysed by I.C.P..

Alcoa uses three procedures. For soda, calcia and iron a sealed tube digest at 230°C for 12 hours using 80% hydrochloric acid is used. The resulting solution is analysed using A.A.S.. For titanium the same digest is used, however a yellow complex is formed using Tiron (disodium 1-2-dihydrobenzene-3-5-disulfonate) at pH of 3.8 and the absorbance measured with a UV-visible spectrophotometer at 380nm. For silica a carbonate-borate fusion is used to dissolve the alumina. The 'molybdenum-blue' complex is formed by reducing the oxidised molybdosilicate complex (yellow) which is formed by addition of ammonium molybdate at controlled pH. The absorbance of the complex is measured using a UV-visible spectrophotometer at 815nm.

A summary of the results is given in Table 1. Repeatabilities are given as 2σ (two standard deviations).

3.1 Soda

The soda in the samples ranged from 0.43% to 0.51%. For each sample the Alcoa values were higher, the overall difference being 0.014%. The repeatabilities as determined from the first three samples (analysed on six separate days in duplicate) were 0.006% (Pechiney) and 0.014% (Alcoa). Although the difference is statistically significant, it would require several determinations on the one sample for such a difference to be verified. The exact reason for the difference was not ascertained. The difference is sufficiently small that it compromises neither of the analytical values and, with the good repeatability, the analyses are capable of meeting the smelters' requirements.

Table 1. Chemical Analyses

Sample No		1	2	3	4	5	6	7	8
Soda	Pechiney	0.50	0.48	0.43	0.46	0.43	0.49	0.45	0.45
Na_2O	2σ	0.007	0.008	0.007					
%	Alcoa	0.51	0.49	0.45	0.47	0.45	0.50	0.46	0.47
	2σ	0.010	0.015	0.016					
Iron	Pechiney	120	99	78	82	82	146	147	97
Fe_2O_3	2σ	4	2	2					
ppm	Alcoa	120	93	70	70	70	140	140	90
	2σ	16	10	12					
Silica	Pechiney	157	146	129	110	99	241	255	142
SiO_2	2σ	15	54	17					
ppm	Alcoa	140	120	130	90	90	240	260	150
	2σ	16	24	22					
Calcia	Pechiney	463	378	463	404	419	487	479	395
CaO	2σ	19	22	60					
ppm	Alcoa	440	360	430	380	370	460	460	370
	2σ	8	8	0					
Titania	Pechiney	42	29	26	29	30	53	54	29
TiO_2	2σ	4	2	3					
ppm	Alcoa	30	20	20	20	20	40	40	20
	2σ	0	0	0					

3.2 Other Elements

The iron contents ranged from 0.007% to 0.014%. The Pechiney values were consistently higher than the Alcoa values with the overall difference being 0.0007%. The repeatabilities were 0.0003% (Pechiney) and 0.0012% (Alcoa).

The silica values ranged from 0.009% to 0.026%. The Pechiney values were consistently higher, the overall difference being 0.0007%. The repeatabilities were 0.003% (Pechiney) and 0.002% (Alcoa).

The calcia values ranged from 0.036% to 0.049%. The Pechiney values were consistently higher with an overall difference of 0.003%. The repeatabilities were 0.003% (Pechiney) and 0.0005% (Alcoa).

The titania values ranged from 0.002% to 0.005%. The Pechiney values were consistently 0.001% higher. The repeatabilities were 0.0003% (Pechiney) and 0 (Alcoa). The zero value for Alcoa is because of lower resolution by AAS and rounding off of the numbers.

Overall there is good agreement between the Pechiney and Alcoa values even though two quite different analytical methods were used. All the analyses are capable of meeting the requirements. The differences obtained, although small, were interesting. Overall the Pechiney values were consistently 5-10% higher

for iron, silica, titania and calcia, yet 2% lower in soda. The consistent difference for the elements other than soda suggests that there is a bias between the two methods. Understanding and correcting that bias might be required in the future if closer tolerance of impurities in smelter-grade alumina is required. The generally better repeatability obtained by Pechiney is primarily related to the superior repeatability and resolution of ICP compared to AAS.

4.0 PHASE ANALYSES

Pechiney and Alcoa uses x-ray diffraction to measure alpha alumina and gibbsite. Both use Siemens D500 diffractometers and DACO microprocessors with cobalt radiation and a post diffraction graphite monochromator. Pechiney use a mortar and pestle (automated) to dry grind samples and then manual packs into the holder. Alcoa uses wet (alcohol) grinding in a micronising mill followed by mounting in a press. The data for phase analyses are given in Table 2.

Table 2. Phase Analyses

Sample No		1	2	3	4	5	6	7	8
Alpha %	Pechiney	6.6	5.9	2.8	3.7	<2	4.1	7	2.6
	2σ	1.1	0.9	0.8					
	Alcoa	6.8	6.6	3.7	3.6	0.4	4.4	6.9	3.1
	2σ	0.2	0.3	0.2					
Gibbsite %	Pechiney	0.3	1.2	0.2	0.2	0.2	0.9	0	0
	2σ	0.1	0.2	0.1					
	Alcoa	0.9	2.5	<0.2	0.6	0.2	1.3	<0.2	<0.2
	2σ	0.2	0.4	0					
% Gibbsite by DSC		0.6	2.1	0	0.4	0.1	1.0	0	0

4.1 Alpha Alumina

The alpha alumina content is measured by comparing the intensity of the (012) reflection against that of an alpha standard. The alpha values ranged from 0.4% to 6.9%. There was not a consistent difference between Pechiney and Alcoa with both positive and negative differences being obtained. Overall the Alcoa values were 0.4% higher than the Pechiney values. The repeatabilities were 1% (Pechiney) and 0.25% (Alcoa). The reported values are rounded to the nearest whole integer so effectively there is no difference between the values obtained. The analysis is quite capable of meeting the smelters' requirement. The better repeatability for Alcoa is a consequence of the tighter control over the sample preparation method.

Previous work (e.g. Roach et al 1990) has highlighted some of the issues related to phase analysis by XRD for both alpha alumina and gibbsite. For alpha alumina the (012) peak is not the best peak because of its sensitivity to micro-extinction and other effects which increase the random errors. However, it is the only peak that can be used. Also there is no 100% alpha

standard. Comparison of the Pechiney "Etalon" standard and the Alcoa SRP-A-87 standard gave a value of Etalon of 102.5% alpha when compared with the Alcoa standard using the (012) peak. Although such a difference will not have an effect on alpha alumina analysis in smelter-grade alumina because of the low values, it does highlight that, even for one of the most characterised and commonly used materials in x-ray diffraction, there is no international standard and that the 100% alpha standards for such large alumina companies as Pechiney and Alcoa are different.

4.2 Gibbsite

The gibbsite contents ranged from below the detection limit <0.2% to as high as 2.5%. The Alcoa values were about double the Pechiney values. The repeatabilities were 0.13% (Pechiney) and 0.3% (Alcoa).

The difference in values is a result of both the gibbsite standards used and the sample preparation method. Clearly gibbsite determination by x-ray diffraction, as currently practised, is neither capable of giving consistent data between laboratories nor of meeting the smelters' requirements. The problems associated with gibbsite measurement in smelter-grade alumina by x-ray diffraction have been documented by Roach et al, (1990). Another paper at this workshop further examines this issue (Roach et al, 1993) and recommends differential scanning calorimetry as the best method for gibbsite. DSC data for the samples are also given in Table 2. The values tend to fall between the two sets of data.

5.0 PHYSICAL PROPERTIES

The physical property data are summarised in Table 3.

5.1 Surface Area

Both Pechiney and Alcoa use 70% He 30% N_2 gas mixtures to measure the B.E.T. surface area with a Flowsorb 2300. Pechiney degasses samples for 30 mins at 200°C prior to analysis, Alcoa degasses for 1 hour at 150°C. The surface areas ranged from 53 to 73 m^2g^{-1}. The Pechiney values were generally higher than the Alcoa values, though never by more than 1 m^2g^{-1}. The repeatabilities were similar, ± 1.5 m^2g^{-1}. Such good agreement is as expected when using similar equipment. The analysis readily fulfils the requirements.

5.2 Loss on Ignition (L.O.I.) 300 - 1000°C

Generally L.O.I. is reported as 300-1000°C, however the Alcoa standard method is 300-1200°C. For comparison purposes the 300-1000°C was used. (The 300-1000°C value is normally about 0.13% lower than the 300-1200°C value.) Pechiney uses 5g of sample which is heated to 300°C ± 5°C for 2 hours and then cooled in a desiccator containing silica gel. The sample is weighed, heated to 1000°C ± 5°C for 2 hours, cooled as before and reweighed.

The Alcoa procedure is essentially the same except phosphorous pentoxide is used as the desiccant.

The L.O.I. values ranged from 0.48% to 0.84%. In all instances the Alcoa values were higher, the overall difference being 0.10%. The repeatabilities were 0.06% for both Pechiney and Alcoa. The differences obtained are statistically significant.

Table 3. Physical Properties

Sample No		1	2	3	4	5	6	7	8
S.A. m^2g^{-1}	Pechiney	60	54	53	60	73	65	54	56
	2σ	1.7	1.5	1.3					
	Alcoa	59	53	53	59	73	64	53	57
	2σ	1.5	1.2	1.1					
LOI %	Pechiney	0.63	0.63	0.38	0.59	0.68	0.66	0.44	0.46
	2σ	0.08	0.06	0.05					
	Alcoa	0.71	0.69	0.48	0.67	0.84	0.80	0.55	0.54
	2σ	0.06	0.04	0.06					
LBD $g\ cm^{-3}$	Pechiney	0.990	0.980	0.960	0.990	0.950	0.955	0.960	0.960
	2σ	0.008	0.010	0.016					
	Alcoa	0.998	0.983	0.968	0.998	0.968	0.978	0.967	0.969
	2σ	0.008	0.006	0.006					
PBD $g\ cm^{-3}$	Pechiney	1.170	1.130	1.100	1.140	1.090	1.081	1.110	1.080
	2σ	0.028	0.022	0.010					
	Alcoa	1.261	1.231	1.191	1.265	1.171	1.231	1.222	1.182
	2σ	0.006	0.004	0.004					
AI %	Pechiney	4.3	3.6	3.2	3.7	4.4	5.3	5.7	5.7
	2σ	1.2	0.8	0.8					
	Alcoa	6.8	6.8	5.4	7.0	6.1	8.2	6.5	7.1
	2σ	0.4	0.3	0.3					

Smelters would like to know the value of L.O.I. with certainty to within 0.05%; currently neither the Pechiney nor the Alcoa analyses can achieve that. Also the bias between the two sets of results is too large. Consequently further work on this analysis has been undertaken by both Pechiney and Alcoa. The Pechiney work indicates that the temperature control at 300°C, the number of crucibles in the crucible block and the time of cooling all affect the repeatability. Ideally the temperature should be maintained at 300 °C ± 1 °C, no more than two crucibles used and the cooling time to be exactly 10 minutes. Those extra conditions can reduce the repeatability to close to that desired. The reason for the bias has not been confirmed but is most likely a result of different desiccants used.

The manual L.O.I. method is fraught with many potential errors because of the rapid weight gains that can occur on exposing the alumina to air. Also furnace temperature control is extremely critical. An alternative method is to use an

automated thermal gravimetric analyser. Alcoa uses a LECO brand for their routine analysis. The values obtained are affected by ramp rate, the degree of sensitivity used, purge gas (if any) etc. However, by choosing appropriate conditions a repeatability of 0.03% can be obtained irrespective of the operator. The main advantage is that no cooling is required during the measurements. Indeed, on cooling the weight gain of the sample can be measured and work has shown that weight gains start to occur at temperatures as high as 600°C. Such equipment can give the complete thermal picture for the alumina and has the potential to give faster and more repeatable analyses. Little bias between laboratories would be expected using such equipment. Many machines are limited to a maximum temperature of 950°C and would require a small correction to report data at 1000°C.

5.3 Loose Bulk Density (LBD)

The loose bulk densities are measured in a similar way by Pechiney and Alcoa with alumina flowing through a funnel into a cylinder of known volume. The cylinder is then levelled and weighed. Quite different cylinder geometries are used, the Pechiney cylinder being short and wide, the Alcoa cylinder being taller and narrower. The loose bulk densities ranged from 0.950 to 0.998 g cm^{-3}. The Alcoa values were always higher, generally by 0.010 g cm^{-3}. The repeatabilities were 0.010 (Pechiney) and 0.008 (Alcoa). The small but statistically significant difference arises because of the different cylinder geometries. More compaction would be expected with the Alcoa equipment and consequently a higher bulk density as was obtained. Standardisation of the geometry would be ideal. As the tests now exist, the agreement is sufficient for smelter requirements.

5.4 Packed Bulk Density (PBD)

Pechiney use the same cylinder as for LBD, the cylinder being tapped from below which helps compact the sample. The level of tapping is sufficient that the whole sample is fluidised with each tap. Alcoa uses the same cylinder as for LBD, the cylinder being tapped on the side and alumina continually added to the cylinder until no more can be added.

The values ranged from 1.080 g cm^{-3} to 1.170 g cm^{-3} for Pechiney and 1.171 to 1.265 g cm^{-3} for Alcoa. The repeatabilities were 0.020 (Pechiney) and 0.005 (Alcoa). Clearly there is an enormous difference in the PBD values. This results from the two quite different methods used. The difference highlights the need for careful definition of terminology. Which of the two packed bulk densities is the one required by smelters needs to be established.

5.5 Dry Screen Sizing

Pechiney and Alcoa use standard dry sieving techniques utilising ROTAP equipment. The sieve data are given in Table 4. Overall there is very good agreement except at 75 μm with the difference on average being 4% (Pechiney

higher). For the two most critical sizes there was a difference of about 1.5% at +100 mesh (Alcoa higher) and 0.6% at +325 mesh (Alcoa higher). The repeatabilities were generally very similar and about ± 0.3% at both +100 mesh and +325 mesh. The difference in the data at these two mesh sizes is statistically significant. The repeatability appears adequate for smelter requirement but reduction in the observed bias would be ideal. Both Pechiney and Alcoa standardise the screens before use because considerable differences even in "standard" screens can occur. However, that does not necessarily remove bias between laboratories unless the same material is used for such calibration. There is a need for calibration samples for the alumina industry to overcome this issue.

Table 4. Screen Sizing

Sample No	1	2	3	4	5	6	7	8
+150μm Pechiney	5.0	4.9	4.0	5.0	3.5	2.6	2.7	4.6
2σ	0.9	0.3	0.3					
Alcoa	6.6	7.0	5.6	6.6	4.7	3.5	3.6	6.6
2σ	0.1	0.2	0.3					
+106μm Pechiney	40.1	45.4	41.9	48.2	40.4	24.9	27.2	43.6
2σ	1.2	1.0	0.8					
Alcoa	40.9	45.6	42.2	46.2	41.1	26.5	28.1	44.3
2σ	0.3	0.7	1.0					
+75μm Pechiney	76.8	84.4	83.1	82.4	87.4	68.1	70.9	85.9
2σ	1.2	0.7	0.8					
Alcoa	73.4	81.0	79.9	78.6	84.7	62.0	65.1	82.9
2σ	0.5	0.6	0.7					
+53μm Pechiney	87.9	93.7	93.7	89.5	95.9	85.8	87.0	95.0
2σ	0.7	0.6	0.5					
Alcoa	88.2	94.0	94.6	87.7	96.5	86.6	88.0	95.9
2σ	0.4	0.3	0.4					
+45μm Pechiney	91.8	96.1	96.7	92.2	97.8	91.4	91.7	97.3
2σ	1.2	0.2	0.2					
Alcoa	92.1	96.7	97.8	90.9	98.9	92.2	92.9	98.5
2σ	0.3	0.2	0.2					

5.6 Wet Sizing at 20μm

The -20μm sizing has become a major focus of attention over the last several years as it is believed to be a better indicator of an alumina's propensity to dust and its flow properties. The Australian Standards Committee for Alumina has recently adopted a wet screen analysis at 20μm using acetone and electroformed screens (Standards Australia, 1991). That is the method used by Alcoa. Pechiney uses an automated method and screen in water. The data are given in Table 5. The -20μm values ranged from 0.3% to 6.0% with the Alcoa values being consistently higher by, on average, 1.2%. The repeatabilities were

0.6% (Pechiney) and 0.3% (Alcoa). The difference may relate to the medium used for wet sieving rather than a sieve calibration problem. The cause of the difference has still to be determined.

Table 5. Wet Screen and Laser Sizing

Sample No		1	2	3	4	5	6	7	8
Wet	Screen	4.9	2.1	0.4	4.0	0.4	4.4	3.6	0.3
Pechiney		0.6	1.0	0.6					
% -20μm	2σ								
	Alcoa	5.3	2.8	1.0	6.0	0.5	4.7	4.0	0.7
	2σ	0.3	0.2	0.1					
Laser Size	Pechiney	13.5	7.5	4.5	11.8	2.7	12.7	10.3	3.7
% -45μm	2σ	1.1	1.2	1.0					
	Alcoa	10.9	6.6	3.2	13.4	2.3	10.2	8.4	2.4
	2σ	0.3	0.3	0.5					
% -20μm	Pechiney	7.2	3.9	0.5	5.9	0.5	6.0	3.0	0.3
	2σ	0.4	0.2	0.1					
	Alcoa	6.6	3.8	0.8	7.9	0.9	6.0	3.6	0.6
	2σ	0.2	0.1	0.1					
% -10μm	Pechiney	4.6	2.3	0.3	2.9	0.1	3.8	0.9	0.1
	2σ	0.4	0.2	0.1					
	Alcoa	3.3	1.8	0.3	3.0	0.4	2.9	0.8	0.3
	2σ	0.2	0	0					
% -5μm	Pechiney	2.5	1.3	0.2	1.2	0.1	1.7	0.3	0.1
	2σ	0.3	0.2	0.1					
	Alcoa	1.8	1.0	0.2	1.3	0.2	1.2	0.4	0.2
	2σ	0.1	0.2	0					

Although the acetone method is more repeatable it is very operator sensitive and reproducibility data for this method indicate a variability of ± 1% (Standards Australia, 1991). As the -20μm content is normally in the range of 1.5% to 5%, that level of variability is not ideal. The mechanised method used by Pechiney would appear to have many advantages, not only in speed but in reducing operator sensitivity.

5.7 Laser Sizing

The new laser sizers can give very precise data on fines in alumina without any pre-treatment (Scott and Roach, 1990). They enable quick and more detailed information to be obtained than previously possible. The samples were run on a Cilas machine (Pechiney) and a Malvern Mastersizer (Alcoa). The data are given in Table 5 for -45μm, -20μm, -10μm and -5μm sizing. Generally there was reasonable agreement, the largest differences occurring at 45μm. Those differences were similar to the differences obtained on the -325 mesh screen sizings. The absolute values for the laser at -45μm were up to 3% higher than

obtained by dry screening, for two reasons. First, a different size parameter is measured and second, the wet laser sizing washes fines from the surface of coarse particles which are held there by electrostatic attraction and are not measured as fines by dry sieving.

The differences in the values obtained with the two laser analysers are related to both sample preparation and instrumentation effects. The use of standard alumina samples could overcome the majority of the differences such that all laser equipment would give virtually the same values. The extra information obtained for sub-sieve sizes (-45μm), the speed of analyses and good repeatability suggest that laser sizing should become standard at sub-sieve sizes, and perhaps eventually at all sizes. Such laser sizers have been used for many years for process control in the alumina industry - their use in product specifications would be a natural progression.

5.8 Attrition Index (AI)

Pechiney and Alcoa use the method developed at the Alcoa Technical Center which is a modification of the Forsythe-Hertwig method. The Alcoa attrition indices, Table 3, range from 5.4 to 7.1% whereas the Pechiney values are between 0.8 to 3.3% lower. The repeatabilities are 0.9% (Pechiney) and 0.4% (Alcoa). In examining causes for this bias small differences in equipment and operating procedure were noted. The Pechiney orifice is slightly larger (0.4mm) than design (0.381mm) and a constant flow rate is used, not constant pressure as by Alcoa. Pechiney calculate the AI by a mass balance of fines, weighing the fines in the thimble and determining the weight in the bed after attrition. Alcoa measures the +325# before and after attrition. Although mathematically there is no difference in the two determinations, the Pechiney method will be prone to more errors. That is probably the reason for the poorer repeatability. The larger orifice used by Pechiney would be expected to give a lower AI as was obtained. This indicates the need for closely following the method and making exact replicas of experimental equipment when such empirical measures as AI are used.

The standard AI method is a relatively long procedure and prone to many errors. On recombining fines, the fines can ball up or tend to coat and block the finest screens and not be measured. Improvements in the design of the column have been made (Matocha and Crooks, 1987) but there are also potential improvements in the method. Use of laser sizing makes the analysis both easier and gives attrition data for all the size fractions. Such information is produced for breakdown studies in calcination at the alumina refineries and gives much more valuable information than the AI number alone. Such information may be of use to smelters. Alterations in the attrition index method to accommodate laser sizing would appear to be the ideal. For standardisation there is either a need for a standard column to be readily available for purchase or standard samples with known attrition indices are required. The former would be preferred, supplied with a sample of known AI.

6.0 CONCLUSIONS

The analysis comparison between Pechiney and Alcoa of Australia has highlighted the need for frank and open discussions on analytical procedures. It has revealed instances where there has been excellent agreement in data such as for the chemical analyses even though different techniques have been used. For the physical properties and phase analyses the comparison has highlighted the need for recognised standard samples (e.g. alpha and sizing) to remove potential bias. The use of terminology can be misleading such as for packed bulk density where two totally different properties are measured, neither of which might be the most suitable for the customer. Some analyses, such as gibbsite by x-ray diffraction, are not capable of giving accurate information and an alternative technique, differential scanning calorimetry, is required. For others such as LOI the use of new automated equipment could lead to the required level of precision which currently is not obtained as well as giving extra information. The use of laser sizing can give more information on the important fines fraction of the alumina and with greater accuracy and precision than currently obtained if standard samples are available. The attrition index method highlighted the need for very careful attention to equipment and protocol details when measuring such empirical properties. The use of laser sizing could readily extend the analysis to give further useful information to the smelters.

Such analysis comparisons are also a vital precursor for detailed technical discussions on alumina quality and the effect of alumina properties on smelting. Without a common understanding of what property is being measured and the limits of the analysis, it is difficult for detailed discussions on alumina quality between alumina supplier and customer. The analysis comparison has not only helped in getting a mutual understanding and appreciation of the analyses, but also highlighted ways where the analyses can be improved. Those improvements include more rapid analysis, more accurate, and precise and informative data, especially by use of the new equipment available. There is a major requirement for alumina standards. Such standards could be a topic for discussion at this Workshop. The standards could be prepared by an allocated body or produced via alumina producers participating in an international round robin to categorise selected aluminas.

REFERENCES

P. Homsi, "Aluminas for Modern Smelting" 3rd Australian Aluminium Smelter Technology Course, Sydney, pp 474-506, 1989.

G.I.D.Roach, J.B.Cornell, D.Y.Li, B.H. O'Connor, "Quantifying Alumina Phases in Smelter-Grade Alumina by X-Ray Diffraction" 2nd Intnl. Alumina Quality Workshop, Perth, pp 209-223, 1990.

G.I.D. Roach, J. Dunn, "Quantifying Gibbsite in Smelter-Grade Alumina" 3rd Intnl. Alumina Quality Workshop, Newcastle, 1993.

Standards Australia "Determination of Particles Passing a 20μm Aperture Sieve" AS 2879.2-1991.

B.M. Scott, G.I.D. Roach, "Laser Diffraction Measurement of Fines in Smelting Grade Alumina" 2nd Intnl Alumina Quality Workshop, Perth, pp339-354, 1990.

Matocha C.K. and Crooks J.H. "Improvements and Standardization of Attrition Index Test" Light Metals 1987 pp 875 - 879.