AA05 - Some Laboratory Pitfalls in Redside Bayer Chemistry

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Abstract

The vast majority of university students earning a chemistry degree spend very little time on the fascinating world of Bayer chemistry, unfortunately. One of the reasons is probably because it is a constantly evolving and rather complex field, highly dependent on a given plant's liquor properties. From the basics of Bayer liquor potentiometric titration to the counter-intuitive desilication reaction and ubiquitous lime reactions, there are endless possibilities for unexpected behaviors, unknown interferences, and misinterpretations of lab results. This paper will showcase some examples where slight changes in the composition of a plant liquor can impact lab results in the course of a research project on chemical reactions. Also part of the discussion is how some apparently minor modifications in lab procedure can drastically change the obvious result if careful interpretation is not carried out. Intended for the curious developing Bayer chemist to the more seasoned professional, this article contains a series of pitfalls to avoid and tips to remember when dealing with Bayer liquor chemistry.

Keywords: Analysis, Bauxite, Bayer liquor, Bayer process, Chemical reactions.

1. Introduction

Although the first two patents by Karl Josef Bayer [1,2] describing his industrial process to produce alumina from bauxite are 135 years old, the field of Bayer chemistry is still evolving. But while it is a fascinating type of chemistry, not many universities dedicate a significant part of the chemistry curriculum to it, especially in areas of the world where the number of alumina refineries is limited. For many chemists graduating from university, the first encounter with the Bayer process and its family can even be shocking. One rapidly needs to understand that what everybody calls "caustic" is in fact sodium hydroxide, NaOH, even if the concentration is in "grams per liter" written "as Na_2CO_3" or "as Na_2O" (not even using subscript numbers), and not moles per liter like we did learn and use in most chemistry classes. Some things can also be misleading, such as the well-known term "causticity" [3] that one could automatically refer to as "caustic concentration", until they realise that there's more than meets the eye in this obviously simple terminology.

The young aspiring Bayer chemists then learn that it is not alumina that comes out of the plant, it is "hydrate", and after asking a few questions they understand that people refer to alumina **tri**hydrate, $Al_2O_3 \cdot 3H_2O$, that will go to calcination to "lose water". Another reason to read and understand that it is dehydroxylation that takes place in the calciner to obtain alumina [4] from aluminium trihydroxide ($Al(OH)_3$), and not dewatering, dehydration or H_2O evaporation.

Still on the topic of alumina in solution, most of the time reported in g/L Al_2O_3, chemical accuracy will tell that there is actually no alumina in solution, the dissolved species being effectively "sodium aluminate", i.e., $Na^+[Al(OH)_4]^-$ (and not $NaAlO_2$). This is where engineers and chemist need to agree on what makes sense and what is practical in the plant on a day-to-day basis.

It usually doesn't take long to figure out that the "alumina-to-caustic" ratio is an important parameter, although the units are not that straightforward at first sight. With some luck, the company uses only one type of unit in all of its facilities. But if we are in the midst of mergers and acquisitions, where companies use A/C ratio, or molar ratio, or RP, things can easily get lost in translation. And once you believe you understand the subtleties of liquor analysis by titration, you find out that dissolved silica can bias the obtained values [5], and that there are various methods including versions "with potassium fluoride (KF)" and "without KF" [6].

Once the liquor concentration units challenge is sorted out, some counter-intuitive chemical reactions tend to get in the way. A low-silica bauxite that may lead to high-silica pregnant liquor being one example (Figure 1), due to differences in kaolin dissolution and desilication product (DSP) precipitation rates, and seed effects [7,8,9,10].

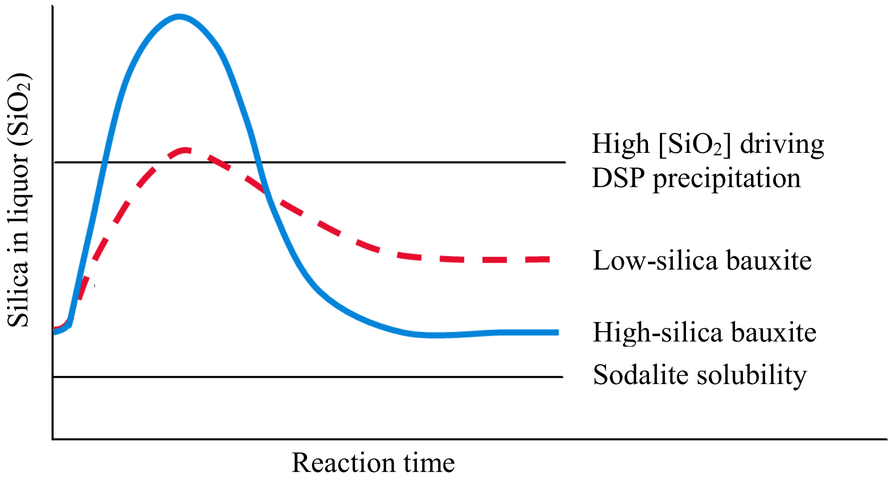

Figure 1. Typical liquor desilication pattern.

But the most striking element remains the ubiquitous calcium: with a solubility of the order of 5 ppm [11], it drives so many reactions and affects so many parts of the process, that lime (CaO) is often referred to as "the *Aspirin*® of the Bayer process" (a well-known pun relating to this drug's developer being Bayer, the Germany- headquartered pharmaceutical firm). Calcium is used from phosphorus control in digestion liquor [12,13], to security filtration filter-aid preparation [14,15], liquor causticisation [16], oxalate causticisation [17], and even fine tuning size control in some precipitation circuits [18]. Calcium is simply everywhere and mastering all the aspects of its chemistry in the Bayer process certainly requires a lot of reading [19,20, among others] and a lot of work.

Fortunately enough, there are a few solid references based on rigorous experimental work, that guide our work in the world of Bayer chemistry, among which Oku-Yamada's silica solubility work [21], Rosenberg-Healy's gibbsite solubility model [22], and many more that can be found in Donaldson's and Raahauge's *Essential Readings in Alumina and Bauxite* [23].

This article is not written as a thorough review of Bayer chemistry but is meant to showcase some laboratory work pitfalls one could encounter when learning to play with Bayer liquor chemistry and related analyses that could lead to misinterpretation of results and be further misleading in developing hypotheses and theories.

2. Experimental Information

A *Metrohm* automatic titrator is used for all Bayer liquor analyses (alumina, caustic, carbonate) in ARDC laboratories. Other analytical work was normally carried out at the Rio Tinto Saguenay Analytical Centre, unless mentioned otherwise.

Digestion work is either carried out using 45 mL Parr® reactors and an electric ALExx rotating block heater, or in an electrically heated 7.6 L Parr® autoclave equipped with two (2) flash tanks in series.

3. Laboratory Pitfalls in Redside Bayer Chemistry

The following sections present a series of examples where lab work can lead to apparently acceptable results, but that careful consideration might change the interpretation. This essentially aims at triggering reflection and discussion among Bayer chemists, both young and experienced.

3.1 Getting Ready to Work with Plant Liquor

A great advantage of working in the lab with refinery liquor, as opposed to synthetic liquor, is that it ensures the presence of the usual impurities. The caveat of that being the variability of the refinery liquor that will be linked to changes in the bauxite feed (composition, blend, etc.) and/or operating conditions. Events such as unit shutdowns or simply extended flowrate reductions can impact spent liquor's impurity content such as silica or phosphorus. A piece of lab work that lasts for many months can therefore be carried out with a series of samples taken in the plant over the course of the project, which could bring variability and require additional precautions. Repeated analyses of the liquor is therefore mandatory prior to each experiment, but sometimes the result may be received too late, i.e. after the experiment is done. This is a "catch 22" type of situation, since some hydrate may start to precipitate out of your spent liquor sample if the delay to get the analytical result is more than a few days. Furthermore, some impurities will precipitate with hydrate, in particular organic molecules, thereby changing, to some extent, liquor chemistry. A good precaution could be to filter the liquor sample as soon as possible, to remove any traces of solids that will act as seed and induce precipitation. Alternatively, keeping the liquor sample "as is" in small-size containers can allow a re-digestion to be carried out prior to the work, to ensure hydrate (and associated impurities) will return in solution.

3.2 Caustic Liquor Carbonation

The reaction of caustic soda with CO_2 from the air (see Equation 1) is well known and is used to induce hydrate precipitation in the nepheline process [24,25], and is part of the Pedersen process concept [26]. It is also a significant source of liquor carbonation in older refineries using air-driven agitation (so-called "air-lifts" or "external draft tubes") for precipitator tanks, or simply having a proportionally high ratio of air-liquor surface of contact to tank volume. For refineries operating a closed residue circuit, i.e. returning water from the residue disposal area, this returning liquor can also bear a high carbonate load due to this same reaction.

$$2\ NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O \tag{1}$$

Fortunately, bauxite's reactive silica and digestion's desilication reaction can handle a significant proportion of the liquor carbonate which will be trapped in the DSP having the generic formula $3[Na_2O\cdot Al_2O_3\cdot 2SiO_2\cdot 2H_2O]\cdot Na_2CO_3$. But liquor carbonation, due to contact with the air can provide misleading results in the lab. Effectively, this carbonation will negatively impact the caustic concentration, thereby artificially increasing the A/C ratio. It is therefore critical to minimise contact between the air and the caustic liquor before and especially after digestion.

An example is shown in Table 1, where two series of experiments were carried out with similar bauxites, but in the first case no special attention was given to air-liquor contact whereas special care was taken in the second set of experiments.

Table 1. Impact of air-liquor contact on values of caustic and carbonate concentrations.

Uncontrolled air-liquor contact			**Limited air-liquor contact**		
Caustic (g/L Na_2CO_3)	**Carbonate (g/L Na_2CO_3)**	**Causticity (C/S, %)**	**Caustic (g/L Na_2CO_3)**	**Carbonate (g/L Na_2CO_3)**	**Causticity (C/S, %)**
221.8	32.8	87.1	221.7	28.2	88.7
220.3	33.3	86.9	219.0	30.2	87.9
219.6	34.0	86.6	220.4	25.7	89.6
216.1	35.0	86.6	218.3	25.5	89.5
212.9	35.9	85.6	218.8	26.5	89.2
212.2	39.7	84.2	218.0	25.9	89.4
213.5	38.9	84.6	217.9	24.6	89.8
212.7	40.3	84.1	217.0	28.7	88.3

The difference can be seen graphically in Figure 2 where carbonation artificially decreases the caustic concentration, directly impacting the A/C ratio. This can lead, for example, to an overstatement of the A/C breakpoint value, by a few percent, in the presence of excessive carbonation, as depicted in Figure 3 (digestion of the same bauxite under the same conditions, with different post-treatment and delays before liquor analysis).

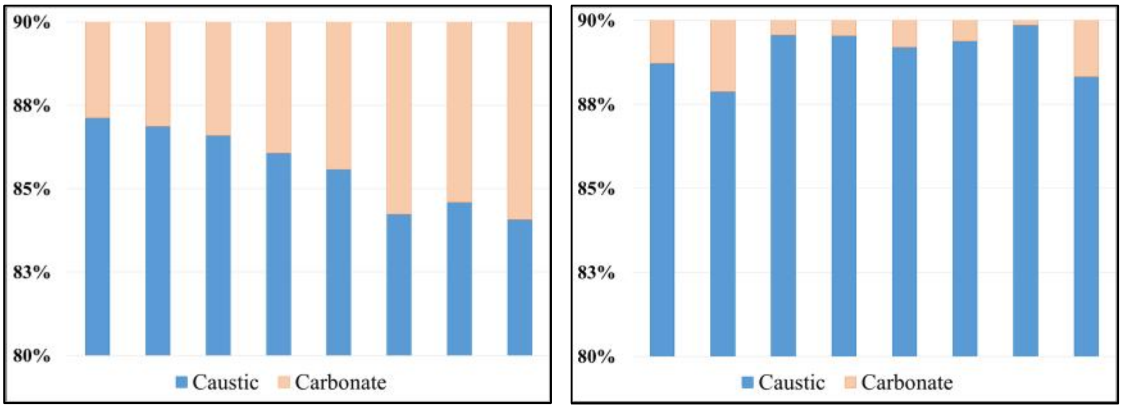

Figure 2. Air-liquor contact impact on carbonation
Left : high air-liquor contact , Right : low air-liquor contact.

Typical precautions that can be used to avoid this type of behavior will be to minimise delay between reaction and analysis, or using watch glass to cover recipients, but also to change the order in which the samples are analysed on the auto-titrator, in particular for lower caustic concentrations which are more prone to carbonation.

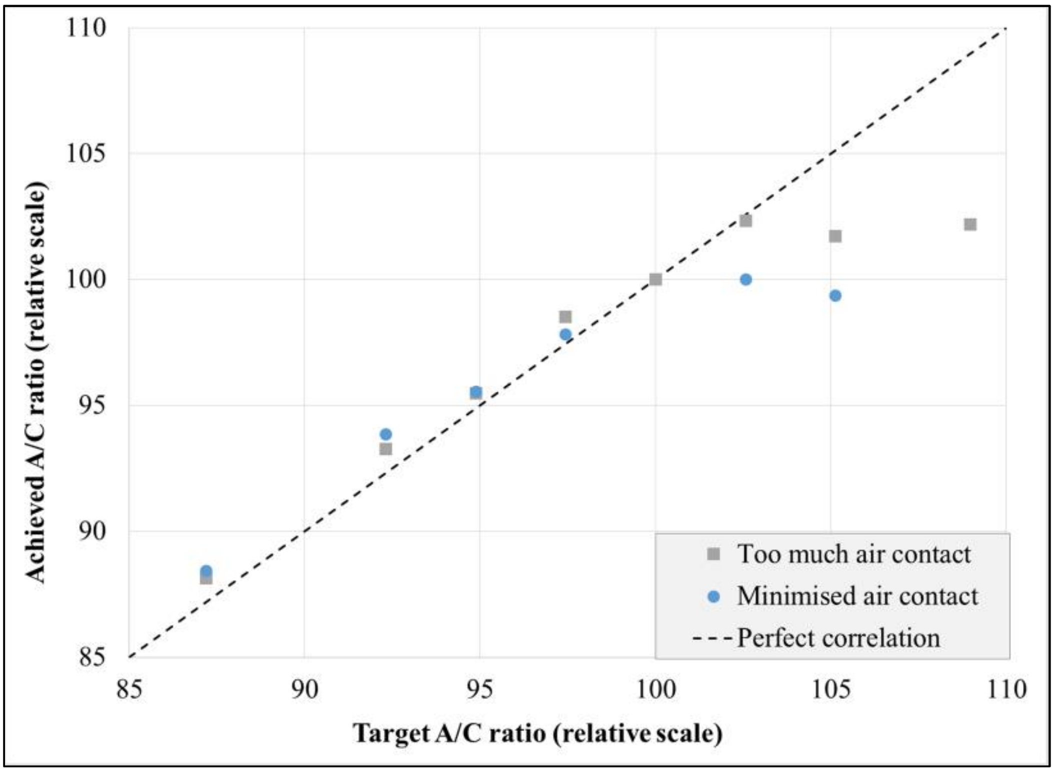

Figure 3. Example of A/C ratio impacted by liquor carbonation.

3.3 Pre-desilication, Digestion, Flashing, Quenching and Reaction Kinetics

Predesilication often requires very long residence times that are not necessarily compatible with a work day in the lab. But undertaking digestion without predesilication, if the refinery of interest operates one, is a recipe for failure in terms of results representativeness. This process step impacts the blow-off slurry properties due to the kaolin dissolution and DSP formation kinetics in predesilication that is different from what happens in a digester at much higher temperatures and solids concentration conditions. The particles formed under so dissimilar conditions will behave differently either because of their specific crystallinity, shape or size, or affinity with flocculants.

Another major difficulty in the lab is to properly simulate digestion equipment and/or digestion process. The double-stream digestion design is especially complicated to reproduce in the lab, as well as representative flash-cooling, and both will impact the slurry behavior in downstream process stages, in particular at settling. And on the other hand, direct cooling instead of flashing will significantly change the pregnant liquor caustic concentration, thereby impacting viscosity, settling and chemistry. There is no easy workaround for this, and comparative testing probably remains the best tool to provide useful results on a relative basis.

Small pressure reactors, such as the 45-mL Parr® ones, are reasonably easy to cool down, for example in an ice-water bath, to remove the pressure and be able to safely open the lid. But depending on the experimental plan, timing of liquor sampling for ICP analysis may not always be the same, and this could lead to surprising differences in silica concentrations, for instance.

Like in the previous example, a low-silica bauxite was first submitted to predesilication and then digested under low temperature conditions. After the prescribed residence time at the digestion temperature, the 45-mL reactors are extracted from the ALExx block heater and cooled in an ice-water bath. In one case, it was just after opening the reactor that a pregnant liquor sample was taken, acidified and diluted to be analysed by ICP. In the second experiment, the reactor was opened, and the content was centrifuged to separate solids from liquor which was left on the bench for 60 to 90 minutes. It was after this "long" delay that a pregnant liquor sample was taken, acidified and diluted to be analysed by ICP. The silica concentrations are shown in Table 2, for four (4) comparable experiments (i.e., same A/C target ratio) but with a "short" or "long" delay between the end of digestion cooling and sampling.

Table 2. Impact of sampling delay on silica concentration of the pregnant liquor.

Short delay	Long delay	[SiO_2] ratio
(g/L SiO_2)	(g/L SiO_2)	short/long
1.76	1.30	1.4
1.85	1.28	1.4
1.90	1.29	1.5
1.91	1.27	1.5

Results clearly show that, in this specific case, the desilication reaction was not complete at the end of digestion which is not such a surprise considering the very low reactive silica content of this bauxite. What was unexpected, however, was that in spite of the relatively low temperature (probably of the order of 60–80 °C) and with very limited solid-liquid contacts following centrifugation, the desilication reaction kinetics was still strong enough to keep pushing the reaction.

The main obvious workaround to avoid this type of situation and surprise, especially with low-silica bauxite, is to first cool the liquor to lower temperatures, and to always use a similar short delay between the end of digestion and pregnant liquor sampling for ICP analysis. That being said, collecting samples at higher temperatures, e.g., 90–100 °C, may reduce the risk of hydrate precipitation. Furthermore, adding liquor stabilisers such as mannitol, gluconate, or tartrate, to a liquor sample, is another safeguard against undesirable precipitation, especially for samples taken in the plant with an unavoidable delay to bring these to the lab for analysis.

3.4 Washing Digestion Solids for Analysis

To properly analyse the bauxite residue solids, and perform various calculations, they must be washed with care at the end of digestion, after the pregnant liquor is removed. The main criteria to meet are to completely remove soluble soda and alumina, not to leach the soda from the DSP, and to minimise losses of solids in the process (so as not to bias analyses and calculations through the loss of specific mineral phases). It is well known that the residual liquor must be removed with care in order to avoid hydrate precipitation in the solids which would bias the composition. An alkaline solution is a good option for the first wash, followed by water. While ammonia (NH_4OH) is alkaline and has many advantages such as no ionic contamination and easy evaporation, its use requires precautions to minimise exposure to it strong odor and irritating vapor. It is often not the preferred option among lab practitioners. In the search for a better option, a method was developed where the first washing step is made with an alkaline potassium chloride (KCl) solution, followed by a couple of water washes. This seems to be the almost perfect solution with tunable alkalinity, and if any KCl remains in the solids at the end, its contribution to the elemental balance (XRF analysis) is easy to overlook since there is usually no potassium in the bauxite or residue.

In some instances, however, an unexpectedly high quantity of potassium was found in the XRF analysis results, which raised some questions and triggered a high-level investigation. The hypothesis was that potassium partially replaces sodium in the DSP composition, since ion mobility in the sodalite cage is a known phenomenon [27], but this was considered unlikely because of the absence of proper reaction conditions, i.e., low DSP solubility and low temperature [28]. Results shown in Table 3 convinced us of the possible K-Na substitution in the DSP, since the residue's potassium (K_2O) content is proportional to the bauxite's reactive silica (r.SiO_2) content in an almost linear fashion ($R^2 = 0.93$, see blue circles in Figure 4). Furthermore, the soda content is too low to correspond to the total DSP expected from the bauxite's reactive silica (r.SiO_2) content, and pregnant liquor silica concentrations indicate that desilication is complete.

Table 3. Potassium and sodium in residue from bauxite with various r.SiO_2 contents.

Bauxite r.SiO_2	Residue K_2O	Residue Na_2O	Pregnant liquor silica
wt %	wt %	wt %	g/L SiO_2
1.0	2.0	0.9	0.9
1.7	3.3	2.0	0.9
2.4	3.3	4.3	0.8
3.1	4.6	6.0	0.9
3.9	6.4	7.7	0.9

From the data in Table 3, one can observe that at higher reactive silica, and therefore higher DSP content in the residue, the extent of K-Na substitution decreases. This is probably due to the limited quantity of available potassium in the rinsing solution or to a faster DSP formation kinetics in digestion leading to a more robust sodalite cage, but this remains to be verified. This is illustrated by the graph of sodium and potassium content in the residue, as a function of the bauxite's reactive silica content, as shown in Figure 4, where the sodium slope is 70% higher than the potassium one. It is worth noting that this graph is shown to illustrate the behaviors in a relative manner and it is highly unlikely that both trends are linear, since the intercepts are different from zero (0) whereas, obviously, a bauxite with 0 % r.SiO_2 should give approximately 0 % alkali content in the residue.

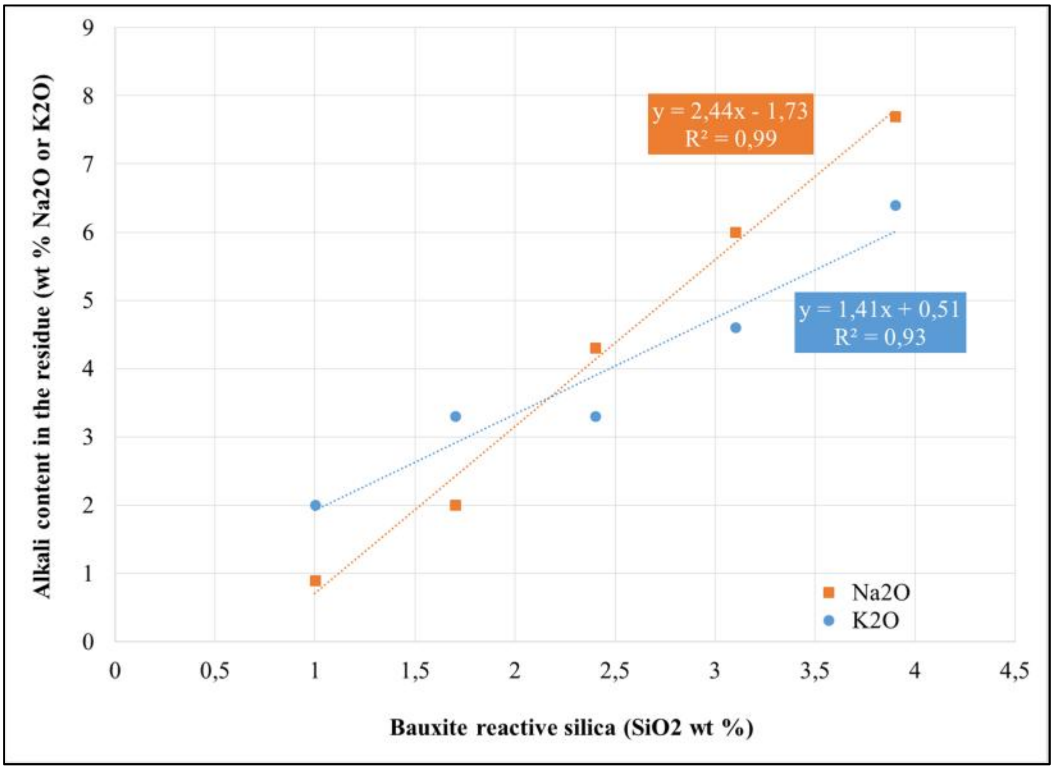

Figure 4. Sodium and potassium in bauxite residue from bauxite with increasing r.SiO_2.

In the end, the critical thing is to use a method that is appropriate to produce results of sufficient quality for further analysis and to understand any potential limitations or impacts the method could have. An initial dilute caustic wash followed by a series of water washes is likely to produce satisfactory results in that respect.

3.5 pH Measurement for Solids

Acid-base reactions and the pH scale are among the first concepts that we learn in high-school chemistry. The Bayer process conditions are at the extreme of this scale due to the high sodium hydroxide concentration of the various liquor streams. Measuring the pH of a solid is, however, not a method that is usually part of the classic chemistry classes in colleges and universities. Fortunately, when it comes to measuring the pH of solidified bauxite residue (filtered, mud farmed, or otherwise consolidated), one can rely on national or international standard methods [29,30,31]. These will be especially useful in the context of bauxite residue valorisation, to determine if the material is submitted to dangerous goods transportation rules based on its alkalinity.

While there are some minor differences between the methods, the basics are all the same, i.e., suspend bauxite residue in water, and measure the supernatant's pH using an electrode and pH-meter. It is well known that obtaining an accurate value is highly dependent on the instrument calibration. This is usually carried out using standard buffer solutions, the most common ones being pH 4, 7 and 10. For a highly alkaline solid, such as bauxite residue, one cannot rely solely on these standard buffer solutions and must use one with a higher pH, such as 12.5 for instance. Effectively, it was shown in our laboratories that a BR sample can give a value of 12.4, when calibrations is done using standards of pH 7 and 10. When adding a pH 12.5 buffer to the calibration, the BR result was found to be 12.6. Such a tiny difference can make or break, when it comes to a legal requirement.

4. Conclusion

This document aimed at providing a few examples of the many unexpected reactions and behaviors encountered in the world of Bayer chemistry. Between a specific plant liquor's unique composition and properties, and potential slips in lab procedures, many things can happen that lead to misinterpretation of results, especially when working in research and trying to develop new areas of knowledge. A rigorous step-by-step execution in the lab is critical and cannot be underestimated when working with saturated or supersaturated solutions like Bayer liquor.

Admittedly in my university years we used to say: "even if one month in the library can save you six months in the lab, six months in the lab is still better than one month in the library". Having said that, the importance of reading technical papers can't be emphasized enough. While there is no "one-stop shop" to learn everything you need to know on Bayer chemistry, the Light Metals series *Essential Readings* [23] is an excellent and highly recommended starting point for younger chemists and engineers, or "returning point" for experienced people looking for a refreshing read.

5. Acknowledgement

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