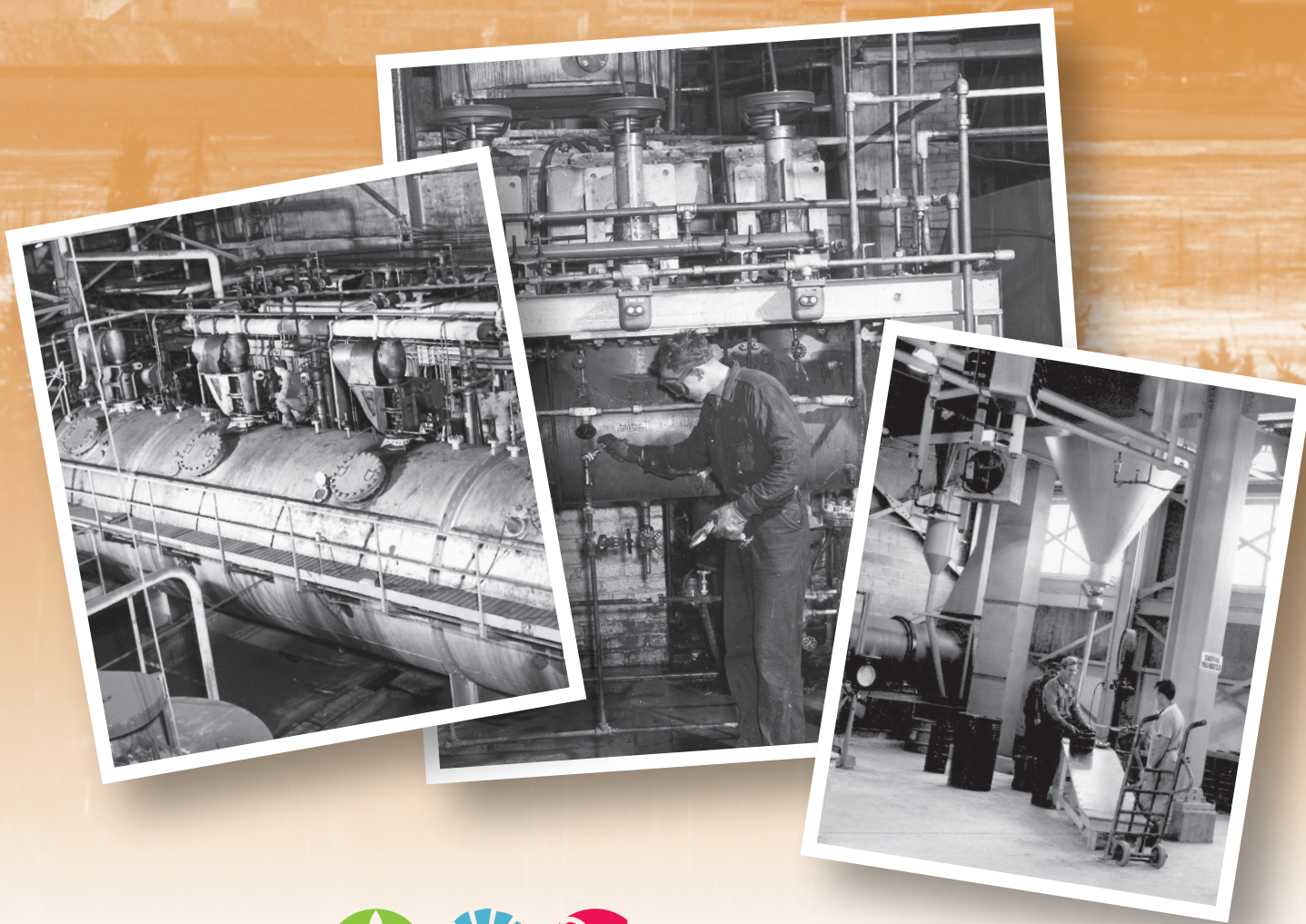


# DEVELOPMENTS IN CANADIAN HYDROMETALLURGY SINCE 1950

Erich Weidenhammer, PhD



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# DEVELOPMENTS IN CANADIAN HYDROMETALLURGY SINCE 1950

Erich Weidenhammer, PhD



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Researcher Neil Colvin takes a sample from a test autoclave at the Ottawa pilot plant during the development of the Sherritt International pressure leach process (Courtesy of Sherritt International Corporation)

CSTM Archives CN005028 Interior of building S6 of the Sherritt Gordon Mines Ltd., where refined nickel is stored

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# INTRODUCTION

Hydrometallurgy is a field of chemical engineering that is primarily focussed on using liquid media to selectively extract metals from their ores. It is usually understood in comparison to pyrometallurgy, which operates at much higher temperatures. Whereas the primary technology of pyrometallurgy is the furnace, hydrometallurgy is associated with aqueous chemistry in equipment such as the autoclave, the stirred tank, and similar vessels used to process liquids and slurries on an industrial scale.

Hydrometallurgy is an “invisible” technology in the sense that it is largely absent from the public imagination. No doubt, many people would be surprised to learn the extent to which metal is produced in chemical plants. Hydrometallurgy is also an important, if little known, theme within the history of Canadian science and technology. The ground-breaking ammoniacal leach process, developed for the Fort Saskatchewan metallurgical facility of Sherritt Gordon Mines during the 1950s, drove international interest in the application of high temperature and high-pressure processes to various areas of mining and metallurgy. Its success provided much of the impetus behind the ongoing legacy of Canadian innovation in hydrometallurgy that is the subject of this report.

Like other aspects of mining industry, hydrometallurgy provides a particularly useful perspective on the shifting circumstances of Canadian economics, research, and culture. It is a technology deployed across the landscape, from the boreal forests of northern Alberta, to the industrial heartland of the Great Lakes Region, to overseas projects processing tropical laterite ores. Its various implementations reveal the aspirations of researchers, business people, and communities both local and national. Sometimes these aspirations were realized, sometimes they were not.

Finally, the technology of hydrometallurgy is profoundly interesting. A hydrometallurgical operation is, to the extent possible, a closed system. This means that inputs and outputs are balanced to ensure an absolute minimum of waste. This produces a fascinating network of interconnections within the operation; chemical reagents used in one stage of the process can be recycled for use in another; residual heat from one operation may be recovered and reapplied elsewhere; a precise amount of one material may be left in solution to facilitate a chemical reaction further down the line. One could compare the development of a hydrometallurgical process to the design of an integrated circuit: in both cases a complex network of interconnections is optimized towards peak efficiency.

# SCOPE AND METHODOLOGY

The goal of this research report is to provide a short and accessible historical account of the development of hydrometallurgical technologies within a Canadian context. It covers six broad themes that have been chosen by a committee of Canadian hydrometallurgists for their importance to the history of Canadian metallurgy.<sup>1</sup> In addition, the documentation and bibliography associated with this report should provide any interested researcher with a solid basis for further investigation.

Most people outside the metallurgical profession would be astonished at the complexity of a typical metallurgical operation. One gains a sense of the intricacy of a hydrometallurgical plant in the following flowsheet of the early ammoniacal leach process for nickel production at Sherritt’s Fort Saskatchewan facility in Alberta.

As this report is meant for a general audience, it cannot provide a comprehensive account of the industrial technologies and the chemical processes they embody. It will instead aim to summarize and to provide relevant anecdotes that illustrate broader issues. Those seeking a greater level of technical detail will find it in the papers, conference presentations, and patents cited below.

Hydrometallurgy is a broad field. The variety of hydrometallurgical techniques and applications ranges from the use of living bacteria in the leaching process, to the use of ion exchange mechanisms to treat mine tailings. One would need several volumes for a full account of Canadian research in this field. However, this research report can provide a broad overview, as well as a good introduction to several of the main institutions, from mining companies, to public and private research institutions.

It must be acknowledged that this short report represents the views and activities of the community of professional chemical engineers working within the field of mining and metallurgy. It relies heavily on the recollections, opinions, and technical expertise of participating hydrometallurgists, and is intended to reflect their view of the field. This approach is not meant to imply that other perspectives and

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<sup>1</sup> The topics for this report were selected by a committee of metallurgists including Jeff Adams, Mike Agnew, Mike Collins, Mike Dry, Dimitrios Filippou, Wenying Liu, Indje Mihaylov, Edgar Peek, Niels Verbaan. James Budac, Mike Collins, Dimitrios Filippou and Indje Mihaylov provided assistance in reviewing the draft. Mike Collins, Mike Dry, David Huggins, Peter Kondos, and Indje Mihaylov provided research interviews for the project. Many others assisted in providing images and permissions. The project was overseen by Mike Dry on behalf of the Metallurgy and Materials Society of CIM, and by Rebecca Dolgoy and Anna Adamek on behalf of Ingenium. Thank you to all involved.

communities are irrelevant to the topic. We currently lack a full body of research on Canadian hydrometallurgy. It is hoped that this report will provide a foundation for a more broadly-based survey of the field.

This account embodies a particular understanding of the recent history of metallurgical research and development that is widely held among metallurgical engineers. For many, the recent history of metallurgical research is the story of a “golden age,” encompassing a period roughly from the 1950s to the 1990s during which circumstances in Canada were favourable to supporting “blue sky” research. Abundant resources were available from government and private sources to develop new metallurgical processes. There is a widespread conviction that a great deal of research capacity has since been lost, to the detriment of Canada’s resource sector. Many metallurgists would like to see a reengagement with that fruitful research tradition, a part of which is documented in this report.<sup>2</sup>

## THE PROCESSES OF HYDROMETALLURGY

It is challenging to describe a “typical” hydrometallurgical process because the possible operations that can be used within a metal processing facility vary tremendously depending on a number of factors. These include the chemical composition of the ore, available supplies of energy and raw materials, neighbouring markets, existing processing facilities, and innumerable others. However, it is possible to describe in very general terms several elements that exist in many hydrometallurgical operations.

In a 1958 article meant to introduce the emerging field of hydrometallurgy to industrial chemists, Professor Frank Forward, a Canadian pioneer within the field of hydrometallurgy and hydrometallurgist of international reputation, listed five main steps in a hydrometallurgical process. Though far from exhaustive, they do provide a succinct introduction to important techniques and terminology within the field:<sup>3</sup>

1) The *Preparation* phase prepares a starting material for the subsequent dissolution of the minerals to be recovered. This usually involves fine grinding to improve contact between the targeted minerals and the leach solution. Other preparations can include flotation, to remove excess material (gangue) and impurities, or roasting, to produce chemical changes that facilitate the dissolution process. On occasion, mining and ore preparation is minimal, and leach solution is injected directly into the ore body, as is the case in some uranium operations.

Pressure oxidation is a preparation process that may or may not incorporate leaching steps. In the processing of challenging “refractory” gold ores, for instance, pressure oxidation is carried out in an autoclave using injected oxygen as an oxidant in order to prepare the ore for subsequent leaching.

2) *Leaching (or Dissolution)* involves the application of leaching solution, or “lixiviant,” to the solid material in order to dissolve the desired material or materials. This process can involve a range of temperatures, pressures, time intervals, and vessels. Leaching can even take place at ambient conditions in

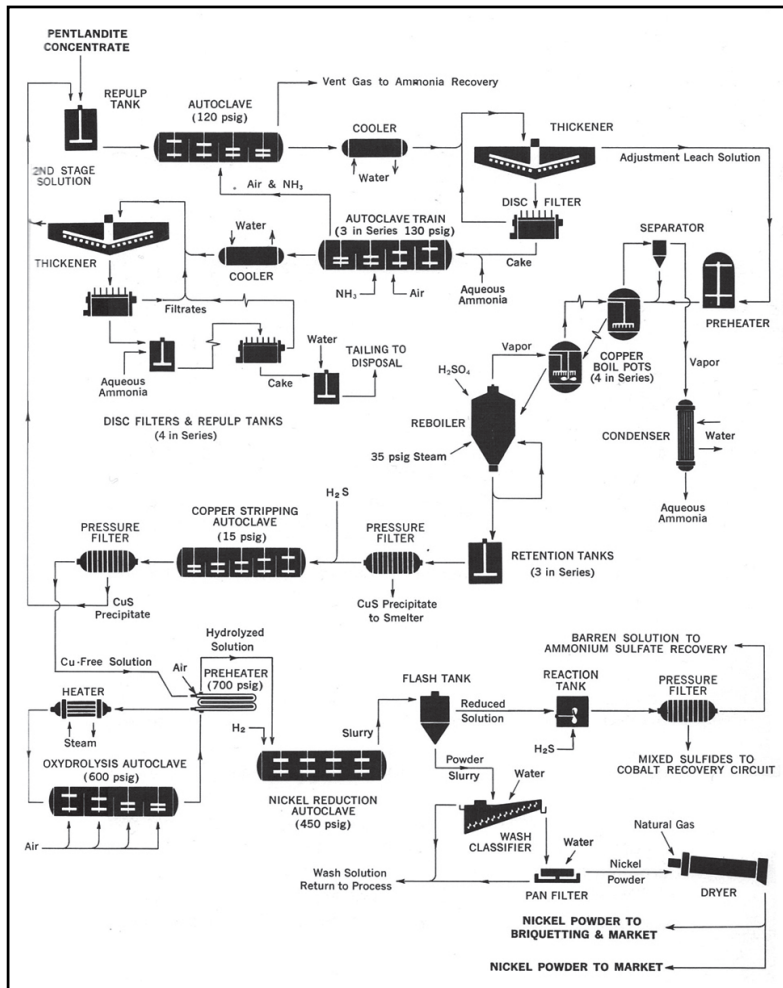


Figure 1: Flowsheet of the Fort Saskatchewan refinery (Boldt 1967, 303).

<sup>2</sup> Kapusta, Mackey, and Stubina 2011, ix-xiii.

<sup>3</sup> Forward 42-45. This list is, to a large extent, a paraphrased version of Forward’s writing, and incorporates quotations.

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the open air, a process known as “heap leaching.” Leaching is often done in multiple stages in order to more efficiently react the feed material with the leach solution as the operation progresses. When the leaching process is complete, the “pregnant” leach solution is recovered from the “barren” solids using processes such as thickening, filtration, and magnetic separation.

- 3) *Purification* of the leach solution is the process of producing purified solutions from which individual metals can be efficiently extracted. This often takes place over several stages that selectively remove other materials that would otherwise negatively affect the final product or lower the efficiency of later steps. Purification might involve the use of a chemical solvent (extractant) to separate a particular metal out of the leach solution (aqueous phase) into an immiscible liquid (an organic phase) that can then be separated away. Dilute leaching solutions—for instance those produced by heap leaching low grade ores—can also be concentrated using processes such as solvent extraction or an ion exchange.<sup>4</sup>
- 4) *Precipitation* is the process of producing a metal, or an oxide powder that can be used to produce a metal, from the purified leach solution. This can involve a number of processes ranging from electrolysis, which deposits very pure metal from an electrolyte solution at the cathode of an electrical current, the addition of chemical reagents to precipitate the final product out of the solution, or the use of a reducing gas under conditions of high temperature and pressure.
- 5) *Regeneration* of the leach solution is often incorporated as a separate step to prepare a portion of the leach solution for reuse.

## PYROMETALLURGY AND HYDROMETALLURGY

The two most common metallurgical approaches are pyrometallurgy and hydrometallurgy. A good way to understand the nature of hydrometallurgy is to compare it to its counterpart and to examine the various factors that determine which approach will better suit a given situation. This comparison is something of an abstraction since, in actual practice, various metallurgical approaches can be combined to process a single ore.

As an example, the Sudbury operation of Vale Canada (formerly Inco) smelts Sudbury nickel concentrate using a pyrometallurgical flash furnace and refines nickel metal using a pressure carbonyl process based on nickel carbonyl vapour (technically “vapometallurgy”). The residue is then treated using a hydrometallurgical process to recover a concentrate of precious metals that is refined elsewhere. Copper metal is recovered from solution through electrowinning, which is technically “electrometallurgy,” but typically falls within the purview of hydrometallurgy.<sup>5</sup>

Nevertheless, in many cases, a mining company will have to decide between a hydrometallurgical route or a pyrometallurgical route when establishing a new facility. The economy of either alternative will depend on a variety of circumstances, including development costs, existing infrastructure, the costs of inputs and the markets for outputs. Meanwhile, evolving regulations limit the nature and quantity of residue outputs, both within a facility, and into the surrounding environment.

The emergence of hydrometallurgy reflects changes within the field of mining and metallurgy that have made existing techniques harder to implement. Pyrometallurgy was traditionally used for rich sulphide ores, which were processed using an evolving family of specialized furnaces. As richer sources have been exhausted, the challenge of processing leaner ores using pyrometallurgy has tended to result in increased energy consumption and pollution.<sup>6</sup>

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<sup>4</sup> Wadsworth 1983, 8; Dry, Mike (June 25, 2018) Personal phone interview. 10:20.

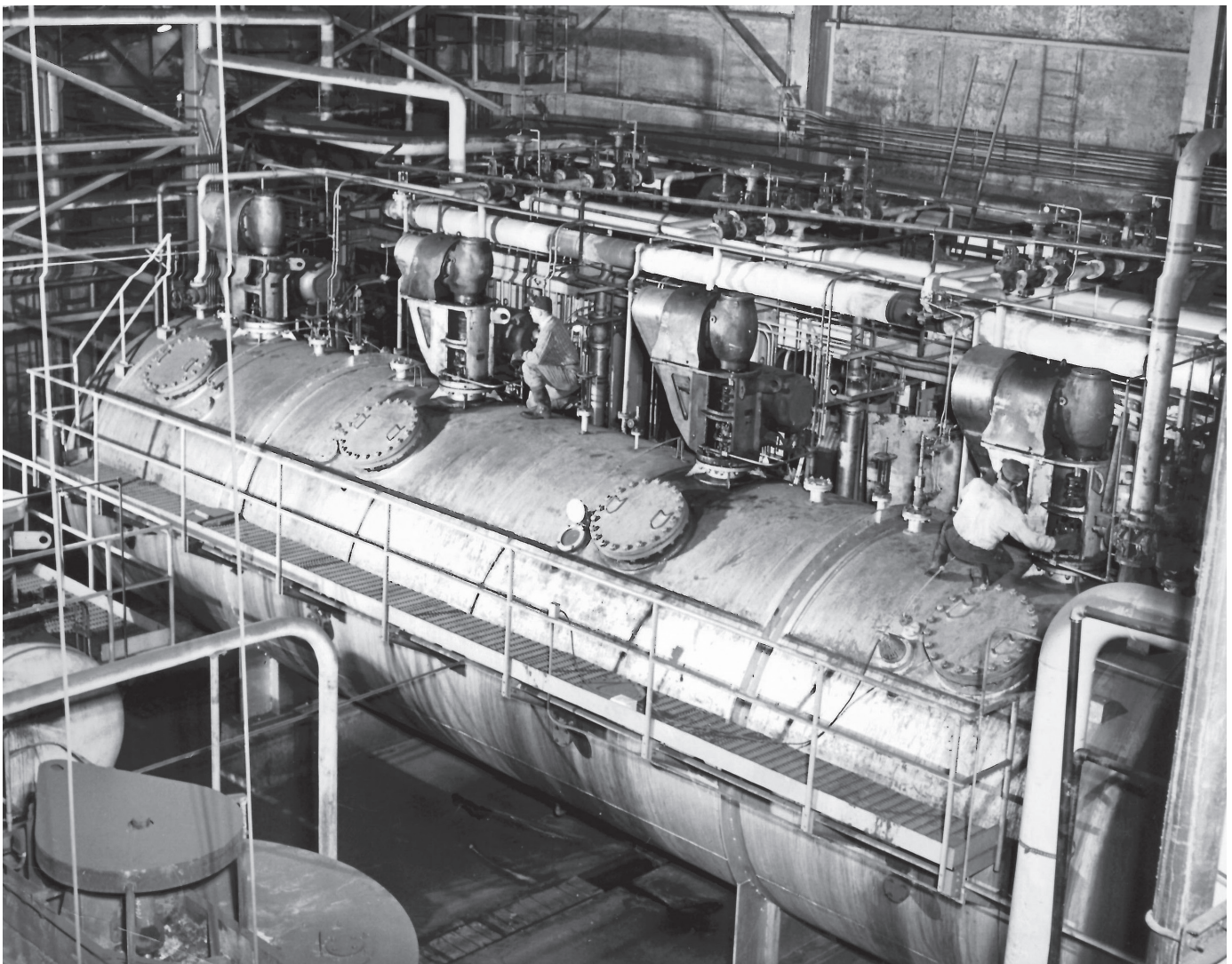
<sup>5</sup> Collins et al. 2011, 299-300.

<sup>6</sup> Habashi 1993, 16.

Pyrometallurgy invests energy into melting comparatively large quantities of material in order to separate the targeted metal out of a molten phase. Hydrometallurgy, by contrast, seeks to selectively dissolve only the economically valuable portion of the ore. This makes hydrometallurgy better suited for processing low-grade ores. For instance, leaching is typically the only economical means to process laterite ores, very low-grade sulphide ores, and for extracting low concentrations of high-value metals such as uranium or rare earth minerals. Processes such as heap leaching, the use of large amounts of low-grade leaching liquors on vast quantities of low-grade ore, can economically recover metal from the low-grade waste rock produced at an existing mining facility.<sup>7</sup>

Pyrometallurgy is typically less sensitive to the specific chemical composition of a given deposit. Hydrometallurgical operations tend to require extensive piloting to determine whether a given process can be applied effectively.<sup>8</sup> On the other hand, hydrometallurgy is better suited to processing very complex ores. Once dissolved into the leaching solution, materials can be recovered sequentially in an efficient, continuous process.

Whether or not one approach is inherently more environmentally sustainable than the other is controversial.<sup>9</sup> Both technologies have evolved considerably in recent years in response to increasing environmental regulation. The inputs and outputs of hydrometallurgy and pyrometallurgy



**Figure 2:** The industrial autoclave, a key hydrometallurgical technology, is shown in this undated photograph from Sherritt International. (Courtesy of Sherritt International Corporation).

<sup>7</sup> Wadsworth 1983, 6.

<sup>8</sup> Forward 1958, 42.

<sup>9</sup> Wadsworth 1983, 6.

tend to differ. Pyrometallurgical processes, which typically take place at temperatures an order of magnitude higher than hydrometallurgical processes, require large quantities of fuel such as metallurgical coal or natural gas.

Hydrometallurgy may require less fuel, but it typically requires large quantities of water. Consequently, the proximity to raw materials may favour one path over the other. Likewise, the residue from pyrometallurgy will tend to go into an exhaust stream, where they will be scrubbed, or into a molten slag, which is relatively inert when cooled. Hydrometallurgy, on the other hand, often produces a concentrated liquid residue that may contain large amounts of heavy metals requiring expensive processing, storage, and disposal.<sup>10</sup>

Hydrometallurgical plants are inherently enclosed systems, meaning that, throughout the entire operation, the various materials flow through pipes into closed vessels. For this reason, hydrometallurgical facilities have been amenable to automation and, hence, have low labour requirements.<sup>11</sup> Early hydrometallurgical plants also avoided problems with fugitive dust and gas then associated with pyrometallurgical facilities.<sup>12</sup> Such advantages have likely diminished over recent decades as upgraded pyrometallurgical facilities have applied advances in sensors, automation, and pollution control. Furnaces, once open to the operator, are now enclosed, so that pyrometallurgical and hydrometallurgical facilities now resemble each other more closely.<sup>13</sup>

One environmental advantage of hydrometallurgy may be found in its capacity to safely process so-called “dirty concentrates” that contain elements that are toxic even in relatively small concentrations. When processed in a furnace, these toxins could end up in the atmosphere and landscape. Using hydrometallurgy, such chemicals can be dissolved into solution and then selectively removed. An example is copper or gold ore associated with arsenic. Using hydrometallurgy, this arsenic content can be concentrated and removed as ferric arsenate, which is stable enough for disposal.<sup>14</sup>

The capacity to more easily produce a greater variety of useful chemical byproducts is a further advantage to hydrometallurgy. This is especially significant given the sulphide minerals that represent the bulk of Canadian ores. In traditional pyrometallurgical process, sulphide ores give off large amounts of sulphur dioxide gas. If released into the open air, sulphur dioxide reacts with water in the atmosphere to produce acid rain.<sup>15</sup>

Pyrometallurgical processes control sulphur dioxide emissions by capturing the gas to produce sulphuric acid, but this is a relatively dangerous chemical that is difficult and expensive to transport and to process. It can be used to manufacture chemical fertilizer, though the market for both sulphuric acid and fertilizer is limited and unpredictable. Alternately, the company could either convert the sulphuric acid to elemental sulphur or neutralize the acid using limestone.<sup>16</sup> Hydrometallurgy offers the possibility of easily producing a wider range of more benign sulphur products, especially elemental sulphur.<sup>17</sup>

These challenges of coping with sulphuric acid were certainly among the key reasons for the development of pressure leaching in Canada. They were summarized in a description of a hydrometallurgical process for copper ore, proposed in the late 1970s, that represents an early effort to transform a large portion of the sulphide mineral into elemental sulphur:

Thus, many Canadian concentrates, notably from the Canadian Shield and the Maritimes, are pyritic or pyrrhotitic [i.e., containing iron], with copper:sulphur ratios worse than the porphyry concentrates that are average for the industry. Mines and many smelters are in remote locations. Major population centers and captive market areas are few and really far between, and cannot support a sulphuric acid consuming industry to anything approaching the extent to which sulphide mining can and must produce sulphur. In many sections of the country, in Manitoba and Northern Ontario for example, suitable limestone for neutralization is unavailable locally and must be brought at high cost. Freight rates for sulphuric acid to almost any destination are high. Phosphoric acid manufacture [for fertilizer production] is handicapped by the need to import high-grade phosphate rock. All these arguments come together with special force for the Canadian non-ferrous mining and smelting industry. Increasingly, and inevitably, it must divert from sulphuric acid to elemental sulphur, which can be stockpiled, sold, or converted to acid as required.<sup>18</sup>

Inevitably, as the overall grade of available ores continues to decrease with time, the relative advantages of the leaching process will continue to grow. Moreover, the demand for high-value rare earth minerals and precious metals, needed in high-technology applications, continues to increase. Finally, new applications continue to emerge within the mining sector, whether driven by regulation, as in the treatment of mining waste, or by the need to optimize existing facilities.

<sup>10</sup> Wadsworth 1983, 6.

<sup>11</sup> Forward 1953, 683.

<sup>12</sup> Forward 1953, 877.

<sup>13</sup> Weidenhammer 2018, 50-51.

<sup>14</sup> Dry, Mike (June 25, 2018) Personal phone interview 6:20.

<sup>15</sup> Habashi 1999, 268.

<sup>16</sup> Kawulka et al. 1978, 133.

<sup>17</sup> Habashi 1993, 16.

<sup>18</sup> Maschmeyer, Milner, and Parekh 1978, 135.

# CONSIDERING THE HISTORY OF HYDROMETALLURGY

The broad history of hydrometallurgy may be approached in a number of ways. It is common, for instance, to search for the earliest origins of a particular phenomenon. In our case, we might look to the alchemical tradition that flourished in the Renaissance and early modern periods. Alchemy was, in many respects, the origin of theoretical chemistry. It posited a fundamental distinction between chemical analysis via the “dry way,” using the heat of the furnace, and the “wet way,” using chemical solvents. Alchemists isolated and identified a number of acids, classifying them by their capacity to dissolve various metals. They also developed chemical means for extracting metals from their ores.<sup>19</sup>

Beginning in the early modern period, one may find various analogies to hydrometallurgical processes. For instance, the heap leaching of copper was practiced beginning in the sixteenth century in areas of Germany and Spain. Copper was leached from copper ore or mining waste using rain and air to oxidize copper. Metallic copper was precipitated from the pregnant solution, which contained copper sulphate, using scrap iron. Similar leaching operations were later used in other proto-industrial processes, such as the production of potash used for the manufacture of soap and glass.<sup>20</sup>

Such an approach to the origins of hydrometallurgy may not be especially helpful. The term “hydrometallurgy” first appeared in *Webster’s Dictionary* in 1864. *Webster’s* dates the first use of “hydrometallurgy” as a means to process metal ores to circa 1859, though it seems to have been used several years earlier in reference to the dissolved metals used in manufacturing processes such as gilding and galvanizing.<sup>21</sup> It then took many decades for this concept to evolve to the point at which it delineated a distinct domain of knowledge covered by a specialized discipline. We should therefore use caution when framing ideas and events using a body of professional knowledge that did not yet exist. Nevertheless, there may be a distinction to be made between a long-lived chemical “art” and a more recent metallurgical “science.”<sup>22</sup>

For a recent example, we can examine the proposed (and ultimately unsuccessful) “Hunt and Douglas process,” which

leached copper using a solution of iron sulphate and brine, and precipitated it using metallic iron. This was patented in 1869 by Canadian James Douglas (1837–1918), and American Thomas Sterry Hunt (1826–1892), both of whom had taught chemistry in Quebec, the former at Morrill College, the latter at Laval and McGill universities.<sup>23</sup>

An early tract on this process, published in 1876, does not mention “hydrometallurgy,” but instead uses the traditional language of “wet processes” and treating ores “in the wet way.” More importantly, their highly empirical approach, their materials, and their tools, were very unlike what would now be recognized as hydrometallurgy. An example:

The copper liquors, whether taken from the stirring or settling tanks, or flowing from the leaching-vats, are received in tanks of any convenient size, where in contact with metallic iron, the chlorids of copper are decomposed, and the copper is precipitated in crystalline grains, plates or crusts, the texture of which will vary according to the strength of the liquors. Wrought-iron precipitates the copper more rapidly than cast iron, but where the latter is the cheaper it should be used.<sup>24</sup>

A more fruitful approach to the history of hydrometallurgy is to trace the gradual accumulation of key industrial technologies. Accounts of modern industrial chemistry typically begin around the emergence of the German chemical dye industry in the late nineteenth century. It was in the German research laboratories such as that of the Bayer firm of Westphalia that the process of scaling-up of laboratory chemistry to industrial operations began to evolve into the profession of chemical engineering. The development of the industrial autoclave was especially important to the eventual emergence of hydrometallurgy. The autoclave is a pressure vessel incorporating mechanical mixers and heating elements for combining chemical reagents on an industrial scale.<sup>25</sup>

The industrial autoclave emerged from the Bayer process of Karl Josef Bayer (1847–1904), which was patented in 1888 to produce aluminum hydroxide as a fixative for fabric dye. This multi-step process involves heating ore in an autoclave with sodium hydroxide to produce aluminum oxide. It remains the standard way to prepare aluminum oxide as feed for aluminum production using the Hall-Héroult electrolytic reduction cell.<sup>26</sup> It is an important, though little known, achievement of Canadian metallurgy that the Sherritt ammonia leach process provided the second application for industrial autoclaves after the Bayer process. The Sherritt process also greatly furthered the development of other key

<sup>19</sup> Brock 2000, 19; 27-28.

<sup>20</sup> Habashi 1993, 4.

<sup>21</sup> “hydro-, comb. form.” *OED Online*, Oxford University Press, June 2018, [www.oed.com/view/Entry/89913](http://www.oed.com/view/Entry/89913), accessed September 4, 2018; “hydrometallurgy” Merriam-Webster online website. <https://www.merriam-webster.com/dictionary/hydrometallurgy> (accessed March 24, 2019); see also: *Humphrey’s Journal of the Daguerreotype and Photographic Arts and the Sciences and Arts Pertaining to Heliography* (1852-1862); New York Vol. 4, Issue 6, (July 1, 1852): 92.

<sup>22</sup> Wadsworth 1983, 4.

<sup>23</sup> Weidenhammer 2018, 52.

<sup>24</sup> Douglas and Hunt 1876, 17.

<sup>25</sup> Brock 2000, 641-644.

<sup>26</sup> Habashi 1993, 6-7.

technologies, such as metal production through hydrogen reduction, that proved critical to the more widespread adoption of hydrometallurgy within the mining field.

Another important development was the invention of the cyanidation process for leaching gold and silver. Applied on an industrial scale at the end of the nineteenth century, the cyanidation process fostered the development of a series of industrial technologies. These include the use of very large reactor vessels incorporating air injection in which the finely ground ore is agitated with the leach solution, as well as filtration plants for purifying the leach solution.<sup>27</sup> Cyanidation produced a massive increase in gold and silver over the first decade of the twentieth century. This long-lived process still accounts for a significant majority of world production of both metals.<sup>28</sup>

A host of other industrial technologies were necessary to make hydrometallurgy available to the mining industry. These accumulated at an increasingly rapid pace throughout the early decades of the twentieth century. For instance, the spread of electrical infrastructure made possible the use of electrolysis to recover metal from leach solution. This was first applied to the production of copper in Chile in 1912.<sup>29</sup> Likewise, the Haber process for producing ammonia fertilizer from atmospheric nitrogen was developed in Germany before the First World War. Besides being among the most important technological developments in human history for its role in increasing food production, it was later applied to the production of leaching agents such as ammonia and nitric acid, thereby increasing the production of metals as well.<sup>30</sup>

The flourishing of industrial chemistry drove a need for industrial chemists. The development of human expertise, in turn, drove a search for innovative processes, making possible the emergence of hydrometallurgy as a scientific and engineering field. Hydrometallurgy emerged as a professional discipline once there were enough people practicing hydrometallurgy—that is, when mining companies and policy makers had become convinced of the need to research the efficacy of hydrometallurgical processes.

This disciplinary perspective is especially important because, while industrial processes had emerged in previous decades to permit the chemical leaching of certain metals, the industry still lacked a sophisticated theoretical understanding of how they worked, and how they could be improved. The emergence of hydrometallurgy as a coherent discipline within chemical engineering provided a sustained focus on analysing and quantifying the underlying chemical reactions so that they could be optimized and adapted to new applications.

<sup>27</sup> Habashi, 16-17, Habashi 1993, 6.

<sup>28</sup> Deschênes 2011, 201.

<sup>29</sup> Habashi 1993, 10.

<sup>30</sup> Habashi 1993, 9; Nashner 1955, 212.

## FOUNDING A DISCIPLINE

In February 1963, the Metallurgical Society of the American Institute of Mining, Metallurgical, and Petroleum Engineers (AIME) held the first International Symposium on Unit Processes in Hydrometallurgy at its annual conference in Dallas, Texas. Reflecting on the recent history of the profession, the conference organizers identified three factors that had contributed to the recent growth of the discipline. These were: The growth of the uranium industry, the development of high pressure leaching and reduction techniques, and “the space age requirements for some less-than common metals.”<sup>31</sup>

If we explore the discipline of hydrometallurgy via its professional gatherings, then we can trace its emergence to the 1960s and through the early 1970s. This coincides with the arrival of the high technology standoff of the cold war, which provided new markets for highly valuable strategic metals.<sup>32</sup> In Canada, defence concerns, most notably the enormous demands of the American defence industry, drove research into the extraction of uranium and nickel using hydrometallurgy. In the latter case, American support for Sherritt’s refinery in Fort Saskatchewan, Alberta, helped to establish a critical centre for Canadian research into hydrometallurgy.

Consequently, the early history of hydrometallurgy likely involves a series of connections between early Cold War research, especially in the area of solvent extraction, and the later adoption of these techniques throughout the mining industry. In Canada, this early history centres on the interrelationship between Eldorado Mining and Refining and the CANMET laboratories of the Government of Canada. The careers and oral accounts of retired Canadian hydrometallurgists, notably Gordon Ritcey and John Dutrizac, would help to unfold this theme. While much of this early history falls outside the scope of this report, interested readers should consult the relevant video recordings of oral interviews conducted through the Mining and Metallurgy Legacy Project.<sup>33</sup>

The 1963 hydrometallurgy meeting in Dallas included a number of Canadian participants from several companies, university mining and metallurgy departments, as well as from the federal government’s Department of Mines and Technical Surveys.<sup>34</sup> In October 1971, the first annual meeting of the Canadian hydrometallurgists was held

<sup>31</sup> Wadsworth and Davies 1963, v-vi.

<sup>32</sup> Wadsworth 1983, 6.

<sup>33</sup> The Mining and Metallurgy Legacy Project is undertaken by the Canada Science and Technology Museum (CSTM) and MetSoc. Video interviews, including interviews of Gordon Ritcey and John Dutrizac (both conducted in February 2016) can be found on the CSTM YouTube channel.

<sup>34</sup> Wadsworth and Davies 1963, vii-vi (“Contributors” section.)

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in Ottawa at the Mines Branch of the Department of Energy, Mines, and Resources. One of the purposes of this meeting was to encourage the formation of an ongoing professional group.<sup>35</sup>

There is some earlier indication of professional organization around Canadian hydrometallurgy. For instance, when the Canadian Institute of Mining, Metallurgy & Petroleum (CIM) re-established a metallurgical section in 1944, it consisted of four technical committees: pyrometallurgy, physical metallurgy, electrometallurgy, and hydrometallurgy. The hydrometallurgical committee was first led by Van H. Smith of British Columbia. Smith had designed a gold cyanidation mill for Cariboo Gold Quartz (CGQ) that opened in 1933 in Wells, British Columbia.<sup>36</sup>

The development of the discipline of hydrometallurgy involved bridging a gap between the mining industry and the growing field of industrial chemistry. In an article written in 1958 and addressed to a readership of industrial chemists, the celebrated Canadian metallurgist Frank A. Forward pointed to a “quasi-parallel development of chemical engineering and hydrometallurgy.” He argued that, popular opinion to the contrary, hydrometallurgy was not, in fact, a branch or species of chemical engineering.

Forward’s main point was this: that the conditions under which the chemical engineer worked were then fundamentally different than those in the metal production industry to which hydrometallurgy was “inseparably associated both in fact and principle.” In the chemical industry, he argued, raw materials were relatively uniform and widely available. The development of chemical processes was largely the province of the research chemist, while the chemical engineer was concerned primarily with developing and optimising physical operations of the plant.

Within the metal production industry, by contrast, the raw materials—reserves of ore secured by a mining company—are both diverse and inherently limited. The hydrometallurgist also played a greater role in developing the chemistry of an operation, due largely to challenges imposed by the complex chemistry of each ore to the efficiency of a given operation.<sup>37</sup>

Forward’s article provides a window on the early circumstances of hydrometallurgy. When he wrote the piece, the field was still subsumed within an existing culture of mining and engineering. Since then, research chemists and chemical engineers have taken on distinct roles within hydrometallurgy. In the 1960s, complex chemical processes such as solvent extraction, which involve sophisticated organic and aqueous chemistry, emerged as a significant aspect of hydrometallurgy. The advancement of chemical theory, and the computational modelling of that theory, has meant that tools and technologies could be shared among scientists working in various chemical fields.

Forward’s views are significant, given his role in developing the process of ammoniacal leaching. Studied in the late 1940s and operational by the mid-1950s, this method was an early and remarkable success within the nascent field of hydrometallurgy. It was also the starting point for commercial pressure hydrometallurgy, and the start of a long and fruitful Canadian research tradition in that field.<sup>38</sup> Finally, the Sherritt project is notable as a successful collaboration between company researchers, university researchers, and research based in government laboratories—a notable product of the richly funded and collaborative research tradition emerging from the Second World War.

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<sup>35</sup> Ritcey 1971, i.

<sup>36</sup> Crossfield 1998, 211; *Canadian Mining and Metallurgical Bulletin*, November 1945, 562.

<sup>37</sup> Forward 1958, 42-43.

<sup>38</sup> Chalkley et al, pt. 1.



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# THE ORIGINS OF PRESSURE HYDROMETALLURGY, AND THE PROCESSING OF NICKEL

The development of the ammoniacal leaching process is among the better documented episodes in the history of Canadian metallurgy, though it is little known outside of the field of mining. This process has been well documented, most notably by Dr. Derek G. E. Kerfoot, who was deeply involved in further development of pressure oxidation processes at Sherritt. What follows is based largely on his account.

One challenge in describing the ammoniacal leach process is that it has been applied to a number of different ores, and has been modified as the feed material to the Fort Saskatchewan plant has changed. Below is a very simplified account of the process as applied to the original nickel-copper pentlandite ores of the company's Lynn Lake mine. A detailed representation of this process can be found in the flow sheet diagrams of Figure 1.

The Sherritt process uses strong aqueous ammonia and atmospheric air to leach nickel and copper, as well as cobalt and zinc, into solution. This takes place in a two-stage process at temperatures of 71–88°C and pressures of 100–150 psig (689–1,034 kPa) in horizontal autoclaves.<sup>39</sup> During this initial leaching process, which takes less than 24 hours, iron in the ore is converted to iron oxide and removed with the tailings.<sup>40</sup>

The partially leached concentrate is thickened and filtered, with the solids passing through a second leach consisting of three autoclaves. The pregnant solution is transferred to a copper removal stage where it is boiled at 121°C (150 °F) using steam injection in order to recover part of the ammonia and to precipitate the copper content as copper sulphide.<sup>41</sup> The

success of this step depends on precisely regulating the ratio of unsaturated sulphur to copper in the previous leaching stage. Once filtered, the solution goes through an additional copper-removal step in a copper-stripping autoclave. The resulting solution is virtually free of copper.<sup>42</sup>

The solution then goes to an “oxydrolisis” step in which sulphate compounds, which would otherwise lead to sulphur contamination of the nickel product, are either destroyed or converted to more stable sulphate ions.<sup>43</sup> Oxydrolisis, and the subsequent nickel reduction (hydrolysis) stage, is done at high temperature and pressure, with the former reaching 246°C and 700 psig (483 kPa). The solution then passes to the nickel reduction phase.

Nickel reduction takes place incrementally over several batches. An initial batch is heated at high temperature and pressure in a hydrogen atmosphere to permit nickel in the solution to form nickel metal around nucleating particles. These nucleating particles are attained primarily by stripping the nickel plating, which was formed during the previous reduction batch, from the walls of the reduction vessel. Once the particles formed in the initial nucleating batch are allowed to settle, subsequent batches precipitate nickel onto the existing particles in a sequential “densification” process that is completed once the particles reach the desired size.<sup>44</sup>

The nickel solids are densified to a slurry and washed. The nickel powder can be desulphurized and sintered into briquettes using a high-temperature process, or sold as powder of various commercial grades. Granular ammonium sulphate, a byproduct of the process produced in massive quantities, has a variety of chemical uses, especially as a chemical fertilizer. Ammonium sulphate from Fort Saskatchewan is typically shipped by rail to the Port of Vancouver for distribution to the world market.

The small amount of cobalt present in the Lynn Lake ores initially remained primarily in the leach liquor. A small refinery to recover this cobalt for resale began operating at Fort Saskatchewan in 1955. Significant further developments in cobalt recovery and production occurred over the life of the plant, especially as it switched primarily to concentrates from Cuba that were much higher in cobalt content.

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<sup>39</sup> Bolt 1967, 299.

<sup>40</sup> Nashner 1955, 217.

<sup>41</sup> Forward 1953, 679.

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<sup>42</sup> Nashner 1955, 217-218.

<sup>43</sup> Forward 1953, 682.

<sup>44</sup> Nashner 1955, 220.

## THE DEVELOPMENT OF SHERRITT'S AMMONIACAL LEACHING PROCESS

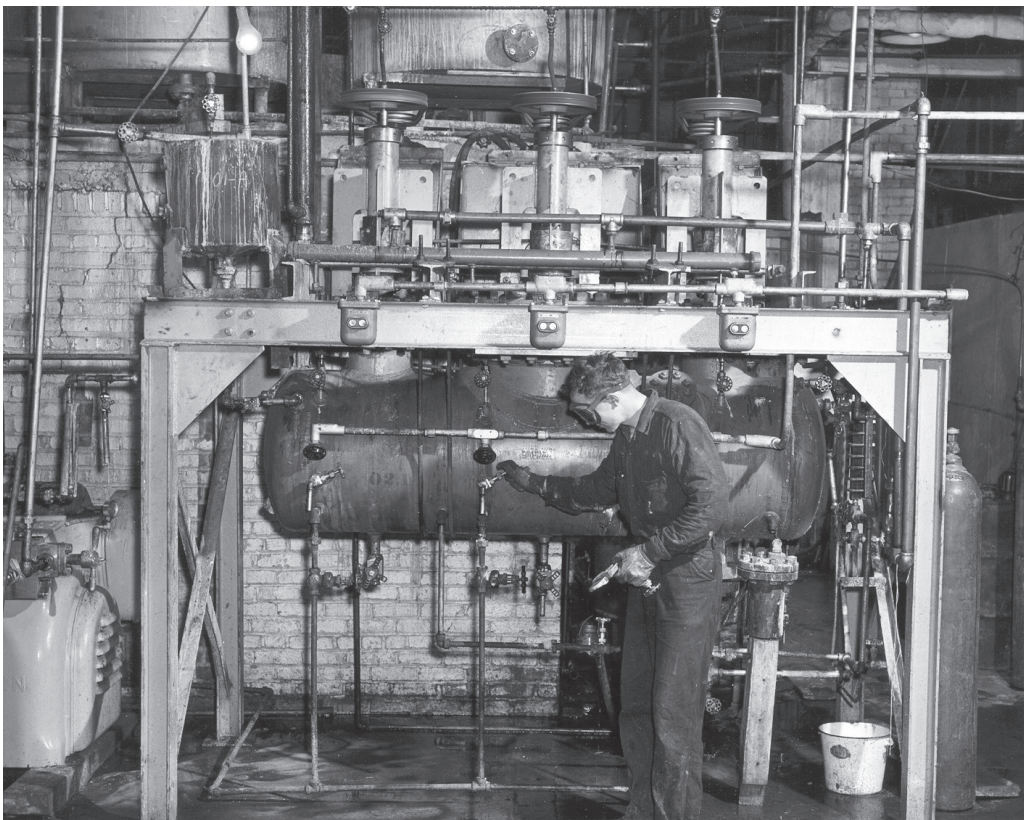
The story of the Sherritt ammonia process begins in the late 1930s, when Toronto-based Sherritt Gordon Mines began prospecting for mining properties beyond its existing copper-zinc-gold mine at Sherridon in the Cold Lake area of northwestern Manitoba. In 1941, a Sherritt Gordon prospector discovered an outcropping of nickel-copper sulphide near Lynn Lake, approximately 200 kilometres north of Sherridon. Due to the wartime demands on Sherritt's existing mine and workforce, the discovery was not claimed until 1945.<sup>45</sup>

The Sherritt ammonia-based pressure oxidation process was developed during the late 1940s and early 1950s to process Lynn Lake ore to a refined state. This was necessary because the Sudbury-based nickel companies, Inco and Falconbridge, were not willing to process Lynn Lake concentrate at their facilities. The task of developing a means to process the Lynn Lake ore was led by Sherritt's manager, the Toronto-born mining engineer Eldon L. Brown (1900–1998). This project had a particular urgency because

a credible process had to be found before the company's existing copper mine was depleted. The window for finding a solution was around five years, or about half the time normally needed to develop a new metallurgical process.<sup>46</sup>

Initial work to produce a concentrate for testing purposes was done at the company's mill at Sherridon. By 1946, the Sherridon laboratory had established that high-grade concentrate could be produced through flotation. Nickel concentrate was between 10 and 14 percent nickel and 1 and 2 percent copper. Copper concentrate was 30 percent copper and 1 percent nickel. By 1947, the company had determined that the site was sufficiently rich to justify production.<sup>47</sup> With concentrate from Lynn Lake available for laboratory-scale testing, Brown enlisted Professor Frank Forward (1902–1972) of the Department of Mining and Metallurgy at the University of British Columbia (UBC). Forward would later be celebrated for the key role that he played in developing the process.

Sherritt had investigated existing processes for nickel, such as smelting followed by electrolytic refining, as was then practiced by Inco. However, the remote Lynn Lake site did not favour these approaches due to the labour requirements



**Figure 3:** Researcher Neil Colvin takes a sample from a test autoclave at the Ottawa pilot plant during the development of the Sherritt International pressure leach process (Courtesy of Sherritt International Corporation).

<sup>45</sup> Brown 1955, 187-189.

<sup>46</sup> Forward 1953, 678.

<sup>47</sup> Kerfoot 1989, 189.

and the cost of fuel and flux.<sup>48</sup> Also, these processes would have lost much of the cobalt present in the Lynn Lake ore.<sup>49</sup>

Forward had a significant background in hydrometallurgical research, which he had explored at the Department of Mining and Metallurgy. His initial work on the Lynn Lake concentrate focussed on applying the Caron process, which had been developed in the 1920s by the Dutch chemist Martinus H. Caron.<sup>50</sup> This was first implemented to process laterite (oxide) nickel ore at a facility in Nicaro, Cuba, a subsidiary of the Freeport Sulphur Company based in Freeport, Texas. The U.S. government had built this plant in 1942 to meet wartime demand for nickel. Forward had worked with Freeport over several months in 1945.<sup>51</sup>

The Caron process involved an initial pyrometallurgical stage of drying, calcining, and reducing the concentrate with hydrogen. This was then leached in a solution of ammoniacal ammonium carbonate. The solution was then heated to produce nickel and cobalt as a precipitate.<sup>52</sup> Forward's adaptation of the Caron process to the Lynn Lake ore would have involved hydrogen reduction of the nickel-copper sulphide concentrate to form metallic copper and nickel. Following a leach in ammoniacal ammonium carbonate solution, the copper would have been removed by electrowinning, and the nickel precipitated as nickel carbonate. This would be calcined to an oxide and reduced in hydrogen to saleable nickel.<sup>53</sup>

In June of 1948, Forward's team at UBC made an important discovery while attempting to leach metallic nickel from a small amount of furnace matte using an ammoniacal/ammonium carbonate solution in an oxygen atmosphere. They discovered that not only had the metallic nickel dissolved, but the nickel sulphide as well. This indicated that the initial reducing step to produce metallic nickel was unnecessary.<sup>54</sup> When applied to the Lynn Lake ore, it was found that nickel, copper and sulphur dissolved, while the iron would remain as an insoluble ferric hydroxide.<sup>55</sup> Furthermore, the process could be accomplished using aqueous ammonia, rather than the ammonium carbonate of the Caron process. The metals—nickel, copper, and cobalt—could then be precipitated out of the pregnant leach, and the vast majority of the ammonia recycled back into the process. A patent was filed for the new process in November 1948, and was granted in 1951.<sup>56</sup>

## COLLABORATION IN THE DEVELOPMENT OF FORT SASKATCHEWAN

To develop these promising discoveries into a functioning metallurgical plant required the involvement of a number of interests. These included both technical experts as well as those who could provide the money and infrastructure required to build a novel metallurgical facility at a very remote site. Among the first requirements was the engineering know-how to produce a system capable of the high temperature and pressure needed to produce metallic nickel through hydrogen reduction.

In December 1948, Sherritt contacted the Chemical Construction Corporation (Chemico), an American company experienced in developing hydrometallurgical projects. Chemico was then studying the pressure leaching of sulphide ores and had recently developed a new method for depositing nickel metal from ammoniacal solutions.<sup>57</sup> In 1949, Chemico was contracted to design an initial pilot plant. In 1952, after a period of fruitful collaboration through a series of pilot plants, the company was granted the final contract to build the Fort Saskatchewan plant.<sup>58</sup>

Also essential to the development of the process were the resources of existing government research institutions. These had emerged over the early decades of the twentieth century to support the Canadian mining industry. With the need for strategic metals during the Second World War, the Mines Branch laboratories had developed world-class research facilities. In June 1949, a team of Sherritt chemists and engineers from Chemico assembled in a building provided by the Mines Branch to begin work on a test plant, the first of several test plants established in Ottawa.

By the spring of 1950, a second pilot plant was established at an abandoned Ottawa foundry. The new plant was to process 272 kg (600 lb) of concentrate per day on a continuous basis. The team expanded significantly as the company worked to develop an increasingly promising laboratory process into a functioning industrial plant. Eldon Brown, reporting on the project, noted that, at one point, the research team included representatives of 20 different nationalities.<sup>59</sup>

<sup>48</sup> Kerfoot 1989, 189.

<sup>49</sup> Forward 1953, 677.

<sup>50</sup> Kerfoot 1989, 189; Caron 1924.

<sup>51</sup> For a detailed discussion of the Caron process at Nicaro circa 1967, see Boldt 425-439; Kerfoot 1989, 190.

<sup>52</sup> Boldt, 425; Kerfoot 1989, 189-190.

<sup>53</sup> Boldt 1967, 190.

<sup>54</sup> Kerfoot 1989, 191.

<sup>55</sup> Kerfoot 1989, 191.

<sup>56</sup> Forward 1951.

<sup>57</sup> Forward 1953, 678; Kerfoot 1989, 191.

<sup>58</sup> Forward 1953, 678; Kerfoot 1989, 193.

<sup>59</sup> Brown 1955, 190.

The hiring of Vladimir Mackiw (1923–2001), a Ukrainian-born chemist, was particularly auspicious. Mackiw was to have a long and productive career with Sherritt, eventually becoming executive vice-president. Among his contributions to the development of the ammoniacal leach process was a method for selectively removing copper as copper sulphide from the leach solution. This made it possible to subsequently precipitate pure nickel.<sup>60</sup>

During this phase, development work on the hydrogen reduction process was transferred from Chemico to the Ottawa laboratory. Chemists from Sherritt and Chemico worked together to address the challenge of nucleating nickel metal during the hydrogen reduction process. Early attempts required the addition of finely ground nickel powder, which was uneconomical. The chemists eventually arrived at a method that used leach solution to dissolve nickel plated on the walls of the reaction vessel during a previous operation.<sup>61</sup> However, the nickel produced in this manner was found to be unacceptably high in sulphur. Further work was required to develop the oxydrolisis step to destroy or convert sulphate compounds to more stable sulphate ions prior to hydrogen reduction.<sup>62</sup>

A third Ottawa pilot plant, with a capacity of 1,360 kg (3,000 lb) of concentrate per day, operated for five weeks beginning in the fall of 1951. Its purpose was to gather operational data for the final refinery, as well as to train a team of about 75 operators and engineers, who would oversee the start-up of the full-scale plant.<sup>63</sup> This was followed by a final refinery built in 1952, which was essentially a smaller version of the final design. By this point, the capacity of the final design had been set at 70,670 tonnes (77,900 tons) of concentrate feed per year.<sup>64</sup>

Over this period, Sherritt transferred its mining and milling facilities from Sherridon to Lynn Lake. This was a massive winter operation involving the transportation of machinery and housing along a 265-kilometre (165-mile) ice road. The undertaking received international media attention. In 1952, the Fort Saskatchewan site was selected for the new refinery due to its proximity to the existing CN railway, which provided a link to the Port of Vancouver. A railway spur to the Lynn Lake site was opened in October 1953.

A final element in implementing the project was the financial and infrastructure support provided by government and business. This was greatly facilitated by the circumstances of the early Cold War. Following an independent engineer's report on the planned refinery, the U.S. government, in cooperation with U.S. steel producers, agreed to purchase the first five years of nickel output from the planned facility, as well as 60 percent of copper and cobalt production. Nickel and cobalt are strategic metals used as an alloying agent in steels with various military applications. The Canadian government, along with the Canadian National Railway, provided the rail link to the Fort Saskatchewan site.<sup>65</sup>

A stock sale, along with various financing arrangements, raised sufficient funds to cover the estimated CAN\$35,029,000 cost of the project.<sup>66</sup> The support of the Newmont Mining Corporation proved pivotal in attracting further investment. Eldon Brown secured this deal with the help of his brother-in-law, who was then Newmont's president.

By 1953, the project was behind schedule and over budget. Estimated costs had increased by a third, driven mostly by the cost of developing the Fort Saskatchewan refinery. The contract with the U.S. government was amended to provide for an advance on part of the metal contract, and an additional financing agreement was arranged with the project's investors.<sup>67</sup> Reflecting on the project's supporters, Brown noted:

Credit for our successful financing must be given to Newmont Mining Corporation, and particularly to Fred Searls, Jr. and Plato Malozemoff, Chairman of the Board and President, respectively, of that company, who had the courage and vision necessary to appreciate the possibilities of a new and radically different treatment process and to convince others of its merits.<sup>68</sup>

Construction of the initial plant at Fort Saskatchewan began in May 1952, and was completed in July 1954. Leaching began in May 1954, with the first nickel metal successfully produced on July 25.<sup>69</sup> By the end of 1954, the plant had reached 90 percent of its design capacity. The following year it reached full operation.<sup>70</sup>

<sup>60</sup> "Vladimir Nicolaus Mackiw (1923 - 2001) Inducted in 2003," *Canadian Mining Hall of Fame* (n. d.) [http://mininghalloffame.ca/inductees/m-of-vladimir\\_nicolaus\\_mackiw](http://mininghalloffame.ca/inductees/m-of-vladimir_nicolaus_mackiw) (accessed 30 January 2016); Kerfoot 1989, 191; Chalkley et al pt 1; Forward 1953, 679.

<sup>61</sup> Nashner 1955, 219-221.

<sup>62</sup> Kerfoot 1989, 192.

<sup>63</sup> Nashner 1955, 223.

<sup>64</sup> Nashner 1955, 212.

<sup>65</sup> Brown 1955, 191.

<sup>66</sup> Brown 1955, 190.

<sup>67</sup> Kerfoot 1989, 192.

<sup>68</sup> Brown 1955, 191.

<sup>69</sup> Chalkley et al pt. 2.

<sup>70</sup> Kerfoot 1989, 194.

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## SHERRITT'S TRANSITION TO MOA BAY CONCENTRATES

While the period following the implementation of ammoniacal acid leach saw Sherritt develop several new processes for other facilities in Canada and around the world, the process used at Fort Saskatchewan remained fundamentally unchanged over several decades. However, by the 1990s, diminishing supplies of sulphide ores and concentrates led Sherritt to modify its leaching process in order to accommodate a newly available and abundant supply of nickel concentrate from Cuba. The cobalt hexamine process is a significant hydrometallurgical innovation.

Sherritt's transition to laterite concentrate, first in Moa Bay, Cuba, later in the ambitious Ambatovy laterite project in Madagascar, also represents political entanglements faced by Canada's large mining companies as they have ventured abroad in search of new sources of ore. While Sherritt's association with a state-owned producer in communist Cuba has placed it at odds with administrations in the United States, the ethical concerns surrounding the mining industry's involvement in the Democratic Republic of the Congo—currently the world's biggest supplier of cobalt concentrate—is no less complicated.<sup>71</sup>

The transition to Cuban Moa Bay concentrate presented a significant technical challenge for the engineers at Fort Saskatchewan. This concentrate, which was especially high in cobalt, was fundamentally different than the previous feed processed at Fort Saskatchewan. A metal traditionally used to produce blue pigments, cobalt has, in industrial times, become an important alloying agent and, more recently, an essential component in lithium-ion battery production. For this reason, it has been an important commodity in the ongoing transition towards renewable energy.

The development and implementation of the Sherritt cobalt hexamine process took place with remarkable speed: less than two years from laboratory testing to commissioning. Laboratory work began in October 1990. By July of 1991,

initial testing had led to a decision to keep the existing ammonia pressure leach process while replacing the nickel-cobalt separation system. The new plant was commissioned in May 1992. An enlarged cobalt-reduction facility, for producing refined cobalt powder, was completed by the end of that year.<sup>72</sup>

The move to Cuban concentrate was dictated by necessity. Sherritt's Lynn Lake mine closed in 1976, after which the Fort Saskatchewan plant operated on concentrates from other mines in Western Canada. In the late 1980s, Sherritt sold its mining assets to become a refinery facility operating on concentrates from Inco's operations in Thompson, Manitoba, as well as concentrates available internationally. By the 1990s, this supply had diminished and the company was faced with the possibility of closing the refinery.<sup>73</sup>

At the time, the Moa Bay plant was among the world's largest suppliers of nickel concentrates from laterite ore. The plant had been originally opened in 1959 by the American-based Freeport Sulphur Company before being nationalized as a result of the Cuban Revolution the following year. With the later collapse of the Soviet economy, and the ending of subsidized fuel exports within the Soviet sphere, Cuban concentrates became available at a time when both Sherritt and the Cuban government were seeking new options.<sup>74</sup> Initial attempts to process Moa Bay concentrate at Fort Saskatchewan encountered difficulties with the high levels of cobalt: 5.5 percent of the total versus .5 percent from Lynn Lake concentrate.<sup>75</sup>

When the existing ammonia pressure leach process was applied to the Cuban concentrate, it was found that iron oxide in the leach residue absorbed cobalt during the initial pressure leach step. This resulted in low cobalt recoveries. Furthermore, in the existing process, cobalt was precipitated from the leach following the production of nickel powder in the hydrolysis step. Because the successful production of marketable nickel required a ratio of nickel to cobalt greater than 20:1, the process was not suitable for high-cobalt feeds. The existing cobalt refining facilities also required significant expansion to accommodate the new supply.<sup>76</sup>

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<sup>72</sup> Kerfoot et al, 148.

<sup>73</sup> Kerfoot et al, 147-148; Kerfoot and Cordingly 1997, 356.

<sup>74</sup> Kerfoot and Cordingly 1997, 356.

<sup>75</sup> Boldt 1967, 301.

<sup>76</sup> Kerfoot and Cordingly 1997, 356; Kerfoot 1995, 3.

<sup>71</sup> See Walt and Meyer 2018.

# THE COBALT HEXAMMINE PROCESS

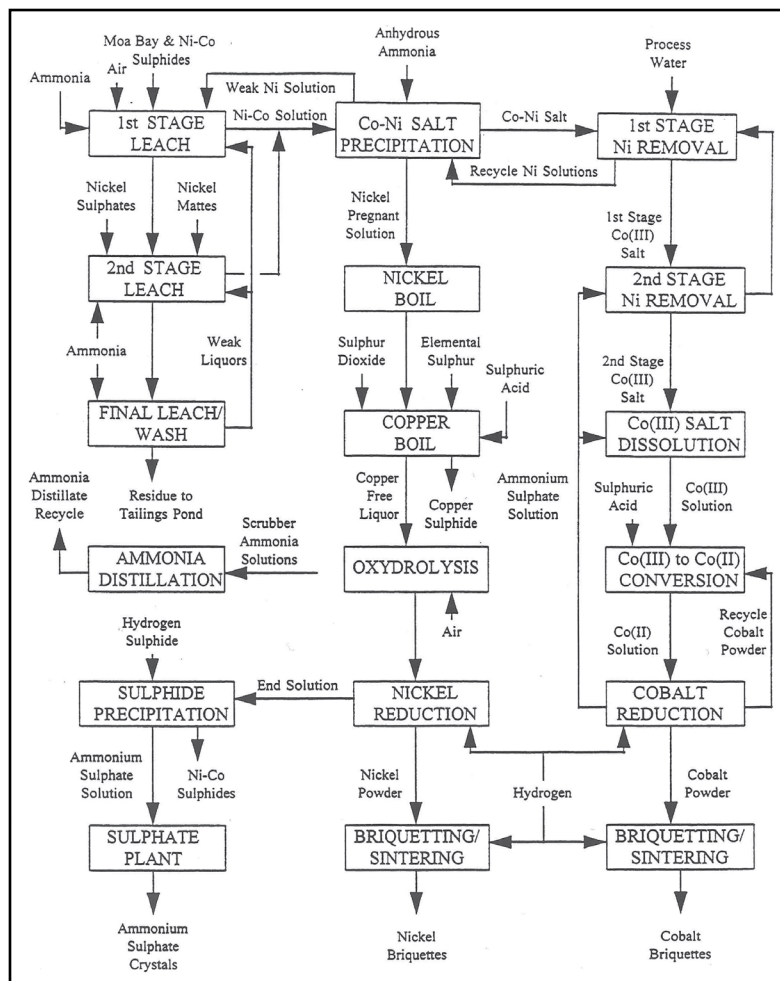
In the modified process, the concentrate is treated as before, using the ammonia pressure leach to dissolve nickel, cobalt, copper, and zinc into solution while precipitating iron and other impurities as hematite. This process is controlled to maximize the formation of hexammine ions of nickel in the oxidation state, and cobalt in the oxidation state. The pregnant leach solution is then filtered and treated with anhydrous ammonia. This precipitates much of the cobalt as a cobalt-nickel hexammine salt.<sup>77</sup>

The cobalt purification proceeds with a leaching step, during which the salt is leached in water and weak recycled leach liquor at ambient temperature, in order to dissolve the nickel portion of the salt. This leaves a cobalt hexammine sulphate salt with a ratio of approximately 100:1 cobalt-to-nickel. The nickel solution is recycled to the salt precipitation process.

The purified cobalt salt is further refined by dissolving it in leach solution, then recrystallizing it by adding ammonia and cooling the solution. This upgrades the salt from a concentration of 100:1 to over 2000:1 cobalt-to-nickel. This is then redissolved and the cobalt is reduced to the CO(II) state through the addition of cobalt powder and sulphuric acid.

The ammonia-to-cobalt ratio is adjusted to 2:1 through ammonia distillation. A final hydrogen reduction step precipitates the cobalt as a metal powder using an autoclave. The cobalt metal is sold as powder or briquettes.<sup>78</sup> Since, in this process, impurities such as copper, zinc, and cadmium follow nickel rather than cobalt, the resulting cobalt salt is very low in impurities.<sup>79</sup>

The nickel solution separated during the precipitation of cobalt-nickel hexammine salt is treated using the ammonia distillation process to achieve a 2:1 ratio of ammonia to nickel. The solution is treated to remove copper, and further purified using an oxydrolisis step. Nickel is then recovered as a metal powder using hydrogen reduction in an autoclave, after which the powder is briquetted for sale. Nickel, cobalt, and zinc remaining in the leach solution are precipitated as a nickel-cobalt sulphide that is recycled to the ammonia pressure leach circuit.<sup>80</sup>



**Figure 4:** Flowsheet of the Fort Saskatchewan refinery (Kerfoot and Cordingley 1997, 358).

<sup>77</sup> Kerfoot 1995, 3.

<sup>78</sup> Kerfoot and Cordingley 1997, 357.

<sup>79</sup> Kerfoot 1995, 3.

<sup>80</sup> Kerfoot and Cordingley 1997, 357.

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## THE CUBAN PARTNERSHIP

The success of this process and the increased availability of Moa Bay concentrate led to incremental improvements in the plant's capacity. In 1994, Sherritt and the supplier of Moa Bay concentrate, La Compania General de Niquel (General Nickel Co SA), which is owned by the Cuban government, created a joint venture called Metals Combined Enterprise. Under this arrangement, Sherritt's Fort Saskatchewan refinery became the exclusive processor of Moa Bay concentrate.

The arrangement was also meant to shield Sherritt from liability related to longstanding U.S. claims on property seized during the Cuban revolution. The deal made Sherritt International the largest foreign investor in the Cuban economy and led to major renovations of the laterite leaching operations at Moa Bay. Sherritt's development work on the leaching process for laterite nickel is an important theme in Canadian hydrometallurgical development, though it is beyond the scope of this report.

Despite these careful legal arrangements, Sherritt fell under the sanction regime imposed on Cuba by the United States. In 1995, the United States formally blacklisted Sherritt for its role in the Cuban subsidiary. Provisions of the Helms-Burton Act targetting the leadership of foreign companies with economic ties to Cuba, still prevent some senior executives at Sherritt International from travelling to the United States.

Cobalt production from Fort Saskatchewan increased significantly following the deal, from an average of 820 tonnes (903 tons) per year in the 1980s, to 2,400 tonnes (2,645 tons) per year between 1995 and 2000. Moa Bay nickel still supplies the vast majority of feed to the Fort Saskatchewan refinery. In 2017, Sherritt produced 1801 tonnes of finished cobalt from Moa Bay concentrate.<sup>81</sup> The cobalt leaching circuit has seen several refinements and upgrades since it was first installed.

The political complexities of processing Cuban concentrate remain a challenge. Following a thaw in Cuban-U.S. relations during the Obama era, the Trump administration has sought to renew the Cuban embargo. In 2018, Panasonic, the exclusive supplier of lithium-ion batteries to U.S.-based Tesla, suspended its relationship with Sherritt due to the presence of Cuban-sourced material in its finished cobalt.<sup>82</sup>

## A RESEARCH TRADITION IN PRESSURE LEACH TECHNOLOGIES

The resources committed to developing the pressure leaching technology at Sherritt ultimately established an important centre for research into pressure leaching at Fort Saskatchewan. Sherritt's capacity to quickly develop a method for processing Moa concentrate was the result of a long period of research and development that led to a series of novel processes. Early in this history, significant efforts were committed to optimizing the leaching process, on fixing mechanical issues not caught during piloting operations, and to developing additions to the plant.<sup>83</sup> Early milestones included a novel cobalt refining process implemented in 1958, and a method for sintering and rolling nickel for use in coinage, which was completed in 1961.<sup>84</sup>

However, at a critical juncture, the company was able to transition from a research focus on optimising its existing process at Fort Saskatchewan, to a much broader effort to bring its expertise in pressure leaching technology to other metals, and to collaborations with other companies. In doing so, it became a rare example of a Canadian mining company that could claim the licensing of technology as a significant revenue stream.

In 1957, Sherritt purchased Chemico's interest in all of the metallurgical patents on which the two companies had collaborated. Continued development on pressure leaching technologies, such as the use of horizontal autoclaves and the nickel-reduction process, would eventually pay off in licensing fees from other mining companies. Over subsequent decades, Sherritt's technologies were applied to a variety of ores and metals, including nickel from laterite ores, zinc, copper, cobalt, uranium, gold, and platinum group metals.<sup>85</sup> Several of these processes will be described in detail over the remainder of this report.

This pivot towards the development of innovative technology came as the result of several factors, not least of which was Sherritt's small size relative to the other Canadian nickel producers. The most significant factor may have been the involvement of a particularly influential chemist, Vladimir Mackiw (1923–2001), who led and championed Sherritt's research group. Michael Collins, current research director at Sherritt Technologies, notes:

Vadimir Mackiw was a real key to stimulating thought starting around the late forties, or about 1950

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<sup>81</sup> Desai and Yamazaki 2018.

<sup>82</sup> Sherritt International Corporation, 2017.

<sup>83</sup> Nashner 1955, 226.

<sup>84</sup> Chalkley et al pt .3.

<sup>85</sup> Chalkley et al pt. 5.

when he joined. ... He gathered and inspired a lot of metallurgists over the next 10 years ... by the time the plant here had been built and operated for a few years, there was really no need for all these technical people anymore, so, essentially, the company recognized the value of this group of people, and they just found other problems for them to work on. That's when they started to look at how they could apply what they had been learning over 10 years together...how they could apply that to other metals.<sup>86</sup>

The first successful collaboration was with Cominco, in British Columbia, which eventually led to the application of pressure leaching to zinc. This was to be the first of a series of pressure leach processes that the company has contributed to the field of hydrometallurgy.

## THE DEVELOPMENT OF INCO'S CRED PROCESS

The Copper Refinery Electrowinning Department (CRED) process was developed at Inco in the 1960s for installation alongside the world's first high-pressure carbonyl nickel refining plant. The carbonyl route to refined nickel, which transforms nickel into tetracarbonyl gas, Ni(CO)<sub>4</sub>, before redepositing it onto a seed material, had first been implemented on a commercial scale at the Mond Nickel refinery in Clydach, Wales, in 1902. (Inco and Mond merged in 1929.)<sup>87</sup> The pressure carbonyl process was developed for Inco's Copper Cliff complex to produce a very high-purity pellet and powder, and to replace its aging nickel refinery at Port Colborne.<sup>88</sup>

The purpose of the CRED plant was to transform the complex residue from this process into a platinum metals group (PGM) concentrate that could be refined at Inco (now Vale's) longstanding precious metal facility in Acton, UK. The CRED facility was commissioned in 1973 alongside the pressure carbonyl plant. Both are still operating essentially unchanged. The Port Colborne nickel refinery was phased out following the introduction of the new facilities, and was finally closed in 1984.<sup>89</sup>

The pressure carbonyl process, and its CRED appendage, were both innovative technologies. From the point of view of hydrometallurgy, the development of CRED represents the early spread of a tradition of pressure metallurgy that had emerged at Sherritt. This technology would flourish at Inco, eventually resulting in the application of pressure hydrometallurgy to enormous plants used to process nickel laterites in Indonesia and New Caledonia, as well as to sulphide ores in Voisey's Bay, Newfoundland.<sup>90</sup>

Inco's pressure carbonyl process was piloted at Port Colborne beginning around 1965. It promised to efficiently recover very pure nickel, but left behind a complex concentrate containing some nickel, all of the copper and cobalt, and all of the company's precious metals. Work began on the CRED process around 1966 under the leadership of David Huggins, then head of hydrometallurgy at Inco. Development continued at Inco's new J. Roy Gordon Research Laboratory in Mississauga, Ontario (now Vale Base Metals Technical Excellence Centre), which opened in 1967.

At that time, much research at Inco had focussed on oxygen pyrometallurgy and the nickel carbonyl process, though the leader of the research and development team at Inco, Walter Curlook (1929-2014), was interested in exploring new areas. In a recent interview, hydrometallurgist David Huggins noted that:

Sherritt Gordon was already doing pressure leaching in Fort Saskatchewan. I would think that what drove me in the direction of doing what I chose to do would have been that as an example.

...I would say that they were the leading face of hydrometallurgy at that time, and I'd like to think that we caught up and ran pretty well, but they were ahead of us.<sup>91</sup>

The development of the process has a notable intersection with research into solvent extraction that was then taking place at the CANMET national laboratory. Based on this ongoing research, a method for separating nickel and cobalt in the CRED process, using solvent extraction in a pulsed column, was designed and built, but never operated.<sup>92</sup> The reasons for this are not clear, though it was much cheaper, and likely less risky, to simply sell the nickel-cobalt concentrate for processing at another facility. In this case, the concentrate was sent to Sherritt's Fort Saskatchewan plant until a new cobalt refinery was built at Port Colborne in 1983.

<sup>86</sup> Collins, Michael. (December 7, 2018) Personal phone interview. 6:00.

<sup>87</sup> Weidenhammer 2018, 80-81; Kapusta, Mackey, and Stubina 2011, 470.

<sup>88</sup> Weidenhammer 2018, 80-81; Boldt 1967, 280-283.

<sup>89</sup> Kerfoot et al. 2011, 129.

<sup>90</sup> Marcuson et al. 2007, 56-70.

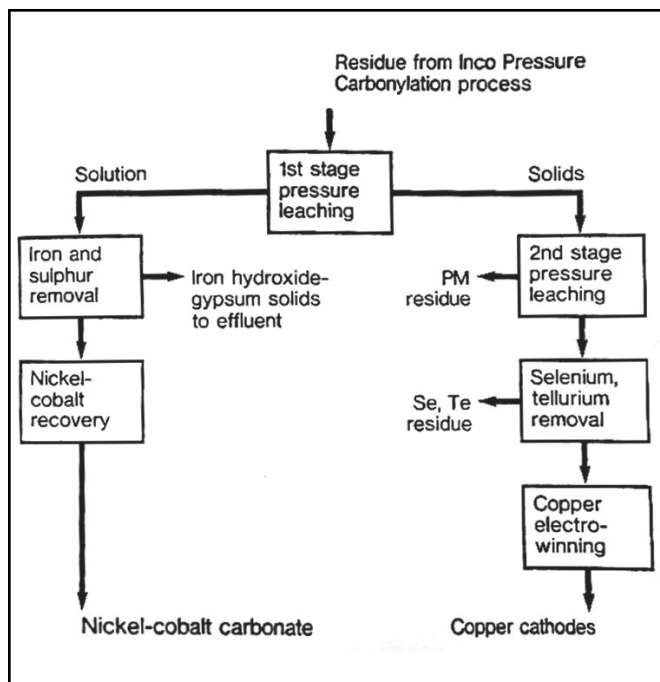
<sup>91</sup> Huggins, David. (January 1, 2019) Personal Phone Interview. 7:00.

<sup>92</sup> Collins et al. 2011, 301, 315-316.



## THE CRED FLOWSHEET

The feed for this process is complex, consisting typically of 55 to 60 percent copper, 6 to 10 percent nickel, 4 to 8 percent cobalt, 4 to 9 percent iron, and 13 to 19 percent sulphur, along with a small amount (622 to 933 grams / 20 to 30 troy ounce) of gold and PGM metals per ton. The main minerals are copper, nickel, cobalt, and iron sulphides.<sup>93</sup> The process has a capacity of 41 tonnes of residue per day.<sup>94</sup>



**Figure 5:** Simplified CRED Flowsheet (Stewart, Tyroler, and Stupavsky 1985, 6).

Residue from the carbonyl plant is treated using an initial pressure leaching step that takes place at 150°C in a leaching medium of sulphuric acid and copper sulphate. This dissolves nickel, cobalt, iron, and arsenic into solution, leaving behind copper, selenium, tellurium, and precious metals. One critical aspect of this initial leach involves consistently matching the correct proportion of copper sulphate to the requirements of a given batch. Early issues with this matching process included copper-depleted solutions attacking the titanium autoclave linings. In the early 1980s, the linings were replaced with brick.<sup>95</sup>

The resulting pressure leach slurry is filtered to separate the pregnant solution from the first-stage filter cake. These go through separate processes, with the liquid entering a series of steps ending in the production of a nickel-cobalt carbonate. The solids are leached to produce a purified cathode copper residue and a precious metals concentrate.

The latter process begins with the solid filter cake, which is mixed with water and charged into an autoclave to undergo a second-stage leaching that takes approximately four to six hours, with some batches taking up to twenty hours.<sup>96</sup> This oxidative pressure leach dissolves copper, sulphur, selenium, and tellurium. The filter cake from this operation is sent for precious metals recovery at Inco's (now Vale's) plant in the UK. The solution contains small amounts of both selenium and tellurium (0.04 to 0.1 grams per litre and 0.02 to 0.06 grams per litre respectively). These impurities must be removed before the solution is processed at the Copper Cliff electro-winning facility.<sup>97</sup>

The selenium and tellurium removal process was altered during the early development of the CRED plant. The earlier process, which was used from 1973 until 1976, employed sulphur dioxide gas as a reductant. This caused problems with plant hygiene as sulphur dioxide escaped from leaking pipes. It was replaced by a system that used two steps: the first involves briefly contacting the leach liquor with metallic copper in a reaction column.<sup>98</sup>

The second step involves aging the solution in a series of four tower vessels over a period of up to twenty-three hours.<sup>99</sup> The residue from this process is filtered and passed to the Inco silver refinery to recover selenium and tellurium.<sup>100</sup> The pregnant solution proceeds to the electro-winning step to produce cathode copper. Copper deposition onto titanium cathode blanks takes seven days.

The pregnant leach liquid filtered from the first stage of pressure leaching contains dissolved copper sulphate that must be recovered in order to prevent contamination of the final nickel-cobalt product. This is done by adding sodium hydrosulphide to the solution in a copper clean-up and precipitation tank at 70°C in order to precipitate copper as copper sulphide. This process must be carefully monitored in order to prevent the excessive formation of toxic hydrogen sulphide gas (H<sub>2</sub>S). The copper sulphide is filtered and returned to the first filtration step in which copper-containing solids are separated from the leach solution.<sup>101</sup>

<sup>93</sup> Collins et al 2011, 299.

<sup>94</sup> Stewart, Tyroler, and Stupavsky 1985, 5.

<sup>95</sup> Tyroler, Sanmiya, and Hodkin 1988, 393.

<sup>96</sup> Saito et al. 1995, 630.

<sup>97</sup> Stewart, Tyroler, and Stupavsky 1985, 10.

<sup>98</sup> Stewart, Tyroler, and Stupavsky 1985, 15-16.

<sup>99</sup> Mokmeli 2016, 205.

<sup>100</sup> Stewart, Tyroler, and Stupavsky 1985, 10-11.

<sup>101</sup> Tyroler, Sanmiya, and Hodkin 1988, 397-398.

Following the copper-removal process, the nickel-cobalt leach liquor enters an iron- and arsenic-removal circuit. Here, in a series of two autoclaves, the pH of the solution is adjusted to 4.1, and the leach liquid processed under pressure in order to precipitate iron as ferric oxide, and to remove arsenic. This is filtered to produce a leach liquid with very low levels of iron and arsenic. Because a significant amount of nickel and cobalt are co-precipitated during this step, the filtrate is repulped with sulphuric acid in water, redissolved, and refiltered to ensure that most of the nickel and cobalt are recovered.<sup>102</sup>

The leach liquor containing cobalt and nickel is sent to the final nickel/cobalt recovery step in which the leach solution is pumped into two reaction vessels into which a sodium carbonate solution is injected. The solution in the first vessel has a pH of between 6.8 to 7.2, while the second is between 8.5 to 9.0. This step precipitates virtually all of the nickel and cobalt. The basic nickel and cobalt carbonates were initially thickened, dried, and shipped to the Sherritt metallurgical complex in Fort Saskatchewan for final processing. Since 1983, they have been processed in a specially built cobalt refinery at Port Colborne.<sup>103</sup>

## THE HYDROMET PROCESS AT THE LONG HARBOUR PROCESSING PLANT

The Long Harbour Processing Plant (LHPP) is a recent, successful, and large-scale Canadian hydrometallurgy project. The plant was constructed to process nickel-cobalt-copper sulphide concentrate from the Voisey's Bay deposit in Labrador. It is located in Long Harbour, Newfoundland, an established industrial site that offers a convenient ice-free Atlantic port, about 100 kilometres west of St. John's, Newfoundland's capital and its largest city.

The Long Harbour plant operates on the hydromet pressure oxidative leach (POL) process. This is typically referred to as the hydromet process. This technology is significant because, while several hydrometallurgical technologies have been developed to process nickel furnace matte, the



**Figure 6:** The dock at Long Harbour where concentrate arrives from the Voisey's Bay deposit (Courtesy of Vale).

<sup>102</sup> Tyroler, Sanmiya, and Hodkin 1988, 399.

<sup>103</sup> Tyroler, Sanmiya, and Hodkin 1988, 400-401.

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Hydromet process produces refined metal from concentrate without need of a smelter.<sup>104</sup> Like the CRED process discussed previously, the Hydromet process was developed at Inco's Mississauga research centre (now the Vale Base Metals Technical Excellence Centre). Its success represents the ongoing contribution of that research establishment. Like the earlier CESL process that is discussed later in this document within the context of copper hydrometallurgy, it is a chloride-assisted leach, though the two processes differ significantly.

The Voisey's Bay mining project, and the Long Harbour plant in particular, can be understood in terms of a longer history of large and costly industrial projects through which the government and people of Newfoundland and Labrador have sought prosperity for their sparsely populated and resource-rich province. The current difficulties surrounding the Muskrat Falls Generating Plant show that this is a continuing trend.<sup>105</sup> An exploration of the Voisey's Bay project reveals the ways in which the legacy of earlier efforts shaped its development.

This project also provides an instructive contrast to earlier cases discussed in this report, notably the Sherritt International metallurgical plant at Fort Saskatchewan. That facility, completed in 1954, had begun production within five years of the opening of its first test plant. Its associated infrastructure, including power lines and transportation corridors, had been built on the basis of a few boardroom agreements. By contrast, the development of the Voisey's Bay project as a whole, and the Long Harbour plant in particular, reveals the challenges that have since emerged for such ventures.

By the closing decades of the twentieth century, large infrastructure projects in Canada had become subject to significantly increased regulation. This included the need to earn the consent of members of the community that had generally been overlooked by planners and policy makers. Whereas economics and technological challenges had once been the primary considerations governing a project's trajectory, these factors were now often dwarfed by the challenge of establishing "social licence to operate" (SLO, often shortened to "social licence"). This concept centres on the need to build community consensus around a project.<sup>106</sup>

The Voisey's Bay project emerged at the confluence of a variety of interests. Foremost was the desire of the provincial

government to secure financial benefits for the province and local communities. Holding a regulatory veto over the project, the province held enormous power in the complex negotiations over where and how Voisey's Bay ore was processed. Also essential to the project's approval was the consent of the Indigenous communities on whose traditional lands the deposit had been discovered. These communities, the Innu and Inuit of Labrador, had long suffered from the indifference of the political class and business community, who had appropriated their traditional lands without consultation or compensation.<sup>107</sup>

Local fishers, homeowners, business owners, environmentalists, and many others have played a role in shaping the regulatory environment, sometimes directly through formal consultation, sometimes indirectly through political influence. The Long Harbour project has been subject to fourteen major pieces of legislation, six federal and eight provincial, which guide the project from planning to decommissioning.<sup>108</sup> Requirements have ranged from estimating the impact on local fish stocks, to ensuring gender equality in the hiring process. Meeting the requirements for the approval necessitated a large contingent of professionals, including negotiators, public health officials, and environmental consultants.<sup>109</sup>

While it isn't possible in the scope of this report to determine exactly how these factors played into the decision to implement a hydrometallurgical process, one can point to various requirements and show how they were met by the process as implemented. As a particular example, we will examine the details of the water supply and effluent disposal system at the Long Harbour site. These provide a contemporary look into a critical element in any hydrometallurgical process: the use of water and disposal of liquid residue.

The Voisey's Bay nickel project encompasses two primary locations, each of which carried particular challenges for the project's realization. The ore deposit was located on the coast of Labrador within the traditional territories of Innu and Labrador Inuit. Additionally, provincial demands that the ore be processed in the province required a suitable site for a major industrial facility. The eventual decision to implement hydrometallurgical process at Long Harbor, Newfoundland was the result of a series of considerations, both practical and political.

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<sup>104</sup> Paul 2011, 22.

<sup>105</sup> Terry 2020.

<sup>106</sup> See Owens and Kemp 2013.

<sup>107</sup> Innes 2001.

<sup>108</sup> Vale Inco Newfoundland & Labrador Limited 2008, Executive Summary, 2-4.

<sup>109</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 11-2, 11-3; Health Research Unit 2007, 1-3;

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## Social licence and Indigenous rights at Voisey's Bay

The lengthy development of the Voisey's Bay project began in 1993, when a prospecting team hired by Diamond Fields Resources discovered a massive nickel-copper-cobalt deposit. A particularly rich part of the deposit, which came to be known as "the ovoid," was understood to be accessible through an open-pit surface mine, greatly reducing the initial mining costs. The deposit's coastal location meant that ore was relatively economical to transport.<sup>110</sup>

The announcement of the discovery generated great excitement within the mining and investment communities. The find ignited a protracted bidding war between the two large Sudbury-based nickel companies, Falconbridge (now Glencore Canada), and Inco (Vale-Inco as of 2006, Vale as of 2010). In 1996, Inco acquired the deposit from Diamond Fields Resources for CAN\$4.3 billion—an unprecedented amount of money that sent the value of Diamond Fields stock skyrocketing. The enthusiasm surrounding the sudden increase in stock value triggered a financial bubble in Canadian mining stocks. This came to an abrupt end the following year, with the notorious collapse of Bre-X Minerals.<sup>111</sup>

Despite the initial excitement, the open-pit mining project quickly ran into opposition on the part of Indigenous and environmental groups. The Innu of Labrador, who know the land as Kapukuanipant-kauashat or Eimish, harbour bitter memories of an earlier industrial megaproject. The Smallwood Reservoir, created as part of the Churchill Falls hydroelectric generating station project of the 1960s, flooded more than 5,000 square kilometres of traditional Innu land. This project, which had been undertaken without consultation, consent, or compensation, is part of a pattern of official neglect that has left the Innu nation of Labrador extremely impoverished.<sup>112</sup>

In the years following the completion of the Churchill Falls project, both the Innu and Inuit of Labrador had emerged as politically organized actors in provincial and federal politics. Land claim negotiations between the Indigenous groups and the Province of Newfoundland were ongoing when the Voisey's Bay discovery took place. Discussions over an Impact Benefits Agreement began with the Labrador Inuit Association in the fall of 1995, slightly later with the Innu Nation. The complex legal challenge of this process involved reconciling the provincial legal framework surrounding mineral claims with the evolving federal land claims process, which increasingly recognized ownership rights of Indigenous groups over traditional lands not covered by the existing treaty process.<sup>113</sup>

Negotiations were prolonged and difficult. Suspicions among environmentalists and Indigenous leaders were

immediately raised over the involvement of mining investor Robert Friedland, the principle shareholder of Diamond Fields Resources. Though celebrated for his acumen as a mining investor, Friedland remains notorious for his involvement in the gold mine run by Galactic Resources in Summitville, Colorado. The mine created a toxic environmental mess when it declared bankruptcy in 1992, abandoning a leaking pond full of cyanide-contaminated leachate. The cleanup has since cost U.S. taxpayers over US\$120 million.<sup>114</sup>

Relations between Indigenous groups and the company reached a low point in 1997 when members of the Innu nation established a protest camp at the mine site to object to activity taking place at the site before a deal had been reached.<sup>115</sup> An agreement was not signed until 2002, when members of the Inuit Association and the Innu nation voted in favour of a proposed deal in the form of an initial "Memorandum of Agreement Concerning the Voisey's Bay Project."<sup>116</sup>

The legal settlement pertaining to Indigenous rights surrounding the Voisey's Bay project involves the interrelated responsibilities of the province and the company (now Vale Canada). The company has negotiated Impacts and Benefits Agreements (IBAs) with both the Innu and Inuit communities. These confidential agreements lay out obligations such as profit-sharing arrangements, hiring quotas, educational and training opportunities, and environmental obligations.<sup>117</sup>

Meanwhile, the company relies on the province to establish legal land ownership and Indigenous property rights through the ongoing land claims negotiations. These, in turn, are connected to broader historical issues of reconciliation and compensation. In 2011, as part of ongoing land claims negotiations, as well as efforts to permit the further development of hydroelectricity, the Innu and the Government of Newfoundland and Labrador signed the "Upper Churchill Redress Agreement," which provided ongoing compensation to the Innu for the loss of their lands.<sup>118</sup>

The environmental assessment for the mine and mill planned for the Voisey's Bay site was completed in 1999. The agreement signed in 2002 permitted construction to begin. The mine and concentrator were completed, and the first ore delivered, in 2005.<sup>119</sup> Between that point, and the opening of the Long Harbour Processing Plant in 2014, Voisey's Bay ore was processed in Inco (later Vale's) existing facilities in Sudbury, Ontario and Thompson, Manitoba. In 2016, construction began on the underground phase of the mine, which is expected to open in 2021 and will extend the mine's production until at least 2032.<sup>120</sup>

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<sup>114</sup> McNish 1998, 22.

<sup>115</sup> Anon 1997.

<sup>116</sup> Goldie 2005, 313.

<sup>117</sup> Natural Resources Canada, 2008.

<sup>118</sup> Landers, 2019.

<sup>119</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 2-2.

<sup>120</sup> Anon 2019.

<sup>110</sup> McNish 1998, 118.

<sup>111</sup> McNish 1998, 321-322.

<sup>112</sup> Innes 2001.

<sup>113</sup> Innes 2001.

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## The Long Harbour facility

The provincial government of Newfoundland (renamed Newfoundland and Labrador in 2001), which controls most aspects of the permitting process, had the most powerful hand in the negotiations surrounding the development of the Voisey's Bay project. The province's role in licencing and enforcing environmental and labour regulations meant that it could block development until its demands were satisfied. Early in the process, Brian Tobin, then Premier of Newfoundland, declared his intention to "gain full benefits from the Voisey's Bay mine, including the construction of a smelter and refinery in our province."<sup>121</sup> The Long Harbour processing plant would not exist without this initial insistence on local processing.<sup>122</sup>

Once again, bitter memories of the Churchill Falls hydroelectric megaproject formed the backdrop to negotiations. Newfoundland had been obliged to rely on the well-established Québécois public hydroelectric utility to complete Churchill Falls because of the province's limited resources and difficult financial situation. Hydro-Québec had assumed the financial risk, and felt entitled to a substantial share of the profits. Negotiations had been fraught, with Hydro-Québec negotiating a fixed-price contract that could extend up to 65 years into the future. As the facility came online in the early 1970s, it became clear that the arrangement was extremely lucrative for Quebec. Despite much animosity, and repeated attempts to overturn the arrangement in court, the deal remains in place until 2041.<sup>123</sup>

Given that experience, Newfoundland entered negotiations with Inco determined not to permit the benefits of processing Voisey's Bay ore to flow through Inco's existing facilities in Ontario and Manitoba. Negotiations began with a meeting between the CEO of Inco and the newly-elected premier of Newfoundland and Labrador, Brian Tobin. During this encounter, a verbal agreement was reached to build a smelter and refinery within the province that would begin operation by 2000.<sup>124</sup> This raised hopes

among communities across the province that they might benefit from new industrial investment.

Initial plans developed quickly. In 1996, Inco announced Argentina, Newfoundland, as the site for the new facility.<sup>125</sup> However, the Asian financial crisis arrived in 1997, driving down demand for nickel. In addition, the deposit proved smaller than anticipated, which meant that a full-scale smelter and refinery operation was uneconomical. Inco sought to renegotiate the agreement based on the new financial circumstances. Inco proposed, for instance, a smelter to process a portion of the ore, and no in-province refining.<sup>126</sup>

Premier Tobin was inflexible. This raised the ire of the Toronto-based mining and business community, members of which were eager to see Inco's investment turn a profit. As negotiations dragged on towards the new millennium, Asian markets recovered, the price of nickel rose significantly, and the two sides drew closer to a deal that would see the construction of an in-province processing plant.<sup>127</sup>

Meanwhile, Inco had begun to examine options for developing a means to process Voisey's Bay ore. Exploratory studies had rejected both pyrometallurgy and bio-leaching approaches. Pyrometallurgy was found to require too much energy and to generate large amounts of emissions. It was rejected over "economic and environmental reasons." Bio-leaching was seen as relatively untested. It also had the potential to create a large amount of acidic effluent, which would raise the cost of storing residue and neutralizing the effluent prior to release to the environment.<sup>128</sup> A chlorine-enhanced leaching approach, however, already explored by hydrometallurgical researchers in various contexts, carried the advantages of rapid and selective leaching.

Development on a hydrometallurgical process for Voisey's Bay concentrate began at Inco's Mississauga research centre in 1999. Patents were filed for this process in 2000, and approved in December 2002.<sup>129</sup>

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<sup>121</sup> Cole 2000.

<sup>122</sup> Love et al. 2002.

<sup>123</sup> Anon 2018.

<sup>124</sup> Goldie 2005, 96.

<sup>125</sup> Cole 2000.

<sup>126</sup> Goldie 2005, 114-115.

<sup>127</sup> Goldie 2005, 132-133.

<sup>128</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 3-2.

<sup>129</sup> Love et al. 2002.



**Figure 7:** POL Process Miniplant at Mississauga, Ontario (Courtesy of Vale).

In 2002, the Government of Newfoundland and Labrador finally arrived at the Voisey's Bay Development Agreement with Inco and the Voisey's Bay Nickel Company. This laid out the key aspects of the project, including the company's labour and environmental obligations. It also detailed the metallurgical facilities involved. Construction at the mine site would include a concentrator plant with a projected cost of CAN\$710 million. This concentrate would be shipped to an in-province refinery.

The 2002 agreement called for an in-province research-and-development program for a hydrometallurgical process that would be implemented at the refinery (referred to as the "processing plant"). This involved the construction of a demonstration plant at a projected cost of CAN\$130 million, to begin operation in 2002. This would test the

feasibility of the hydromet process that had undergone bench-scale testing and process modelling at Inco's research facility in Mississauga.<sup>130</sup> Testing at the demonstration plant provided the foundation for the processing plant, which was projected to begin construction in 2008 at an estimated cost of CAN\$800 million.

The province also sought to ensure that an in-province plant would be built whether or not Inco's hydromet technology proved viable. For this reason, the agreement included a less ambitious "matte plant" alternative under which Voisey's Bay concentrate would be processed at Inco's existing smelters, then returned to an in-province refinery. This plant would have a total capital cost of CAN\$670 million and employ 500 fewer people than the hydromet plant.<sup>131</sup>

<sup>130</sup> Stephens et al. 2009, Slide 5.

<sup>131</sup> Voisey's Bay Development Agreement 2002, 16.

The primary distinction between the hydromet plant and the matte alternative was the concentration of the feed material. The hydromet plant operated on a concentrate, thus entailing larger volumes of input material and greater amounts of residue. Estimated inputs would be 269,000 tonnes of concentrate at 19 percent nickel, 42 percent iron, 2 percent copper, and less than 1 percent cobalt. It used a combination chloride-sulphate leaching medium. The matte route involved an initial pyrometallurgical step producing a substantially more concentrated furnace matte at 54 percent nickel, 19 percent copper, and 1 percent cobalt. This employed a purely sulphate leach to extract the metals from 92,000 tonnes of matte per year.<sup>132</sup>

One significant difference between the two plans was that the hydromet process produced a blended residue by-product. Because this residue would contain a significant portion of elemental sulphur, it would require neutralization, and permanent storage underwater in a tailing pond, to prevent the development of sulphuric acid. The matte plant route would have produced a solid gypsum discharge that could be stored above ground<sup>133</sup>

Initial piloting on the hydromet process began in 2002 in Mississauga, using a 1:10,000 miniplant that produced 20 kg per day of cathode nickel from 100 kg of concentrate. Each process was implemented as separate and interconnected miniature units within the laboratory. This program lasted until 2004. The next phase, a demonstration plant scaled at 1:100, operated at Argentia Bay, Newfoundland, between October 2005 and June 2008. This was a substantial operation, producing 651 tonnes of finished nickel from 3,810 tonnes of concentrate, along with 65 tonnes of copper and 28 tonnes of cobalt.

The purpose of the piloting campaign was to develop the hydromet process into a reliable industrial technology that could safely and efficiently process thousands of tonnes of raw material using potentially hazardous chemicals. In later communications, the testing team listed the following five key technical solutions developed during the operation at Argentia Bay:<sup>134</sup>

- The control of the process water balance via an anolyte bleed stream. This required resolution before the process could be operated continuously.

- An improved method of selectively rejecting magnesium in order to prevent its co-precipitation with nickel and subsequent build-up in the system.
- An improved means of rejecting silica in order to prevent the blockage of filters and anode bags.
- Improved filtration to ensure a clean supply of feed to the electrowinning stage.
- Several improvements to prevent the premature failure of anodes.

The location of the pilot plant was a boon to the community of Argentia Bay. Between 1941 and 1994, it had been the site of Naval Station Argentia, a large American naval base. The closure of this facility had been a major economic blow. In accordance with the agreement between the province and the company, the testing plant was run primarily by a local workforce.

In 2006, however, Inco declared its intention to establish the processing plant at Long Harbour, 15 kilometres distant from Argentia, due to concerns over environmental contamination at the former American base. Another factor may have been the need to transport residue slurry, destined for the residue storage area, through existing settlements had the plant been built at Argentia.<sup>135</sup> This change of plan caused much concern on the part of the province, which sought unsuccessfully to have the federal government exempt Inco from environmental liabilities related to the site.<sup>136</sup>

As an existing industrial site with a history of chemical production, established port facilities, and transport infrastructure, Long Harbour was convenient. Beginning in 1966, it had been the site of the Electric Reduction Company of Canada Industries (ERCO), a phosphorous plant constructed by Albright and Wilson Ltd, a British company. The government of Premier Joey Smallwood (1900–1991) had attracted the company to the site with generous tax incentives. The province also provided quantities of heavily subsidized hydroelectric power needed to produce elemental phosphorous from phosphate rock. The port location permitted the importation of raw materials from the United States.<sup>137</sup>

<sup>132</sup> Vale Inco Newfoundland & Labrador Limited 2008, Executive Summary, 26, 29.

<sup>133</sup> Vale Inco Newfoundland & Labrador Limited 2008, Executive Summary, 10, 15.

<sup>134</sup> Stephens et al. 2009, Slide 23.

<sup>135</sup> Mihaylov, Indje (March 20, 2020) Personal Phone Interview. 23:00.

<sup>136</sup> Anon 2006.

<sup>137</sup> Martin 2006.

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The phosphorous plant had suffered from very poor environmental controls that had resulted in several notorious instances of contamination. In 1969, for instance, local fishers discovered dead fish and crustaceans in the area. This problem was traced to plant effluent. Coke dust from the plant's smokestacks also spread over local communities. The plant's closure in 1989 caused significant harm to the local economy. However, it left behind an established industrial site and a community eager for renewed development.<sup>138</sup>

In 2006, the year that the Argentia pilot plant began operation, Vale approached Sherritt technologies for a collaboration on developing a process for removing cadmium from the nickel raffinate. Through this collaboration, Vale sought to draw on Sherritt's cadmium-removal technology, which had been developed at the Fort Saskatchewan plant. Development and piloting of a process for removing this

impurity took place at Sherritt's laboratories. In 2017, the cadmium-removal circuit was successfully implemented at the Long Harbour Hydrometallurgy Plant.<sup>139</sup>

The success of the piloting campaign at Argentia permitted the company to definitively select the Hydromet process over the matte plant alternative late in 2008. Initial construction work began at the Long Harbour site in February 2009. In 2010, Vale announced a CAN\$10 billion investment in its Canadian operations, part of which was committed to the construction of the Long Harbour facility.<sup>140</sup> In July 2014, the Long Harbour plant produced its first refined nickel. It operated initially with feed comprising matte from Vale's plant in Indonesia, and testing quantities of Voisey's concentrate. In May 2015, it received its first full shipment of concentrate from the Voisey's bay mine. This was approximately 10 years after the mine produced its first concentrate.<sup>141</sup>

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<sup>138</sup> Martin 2006.

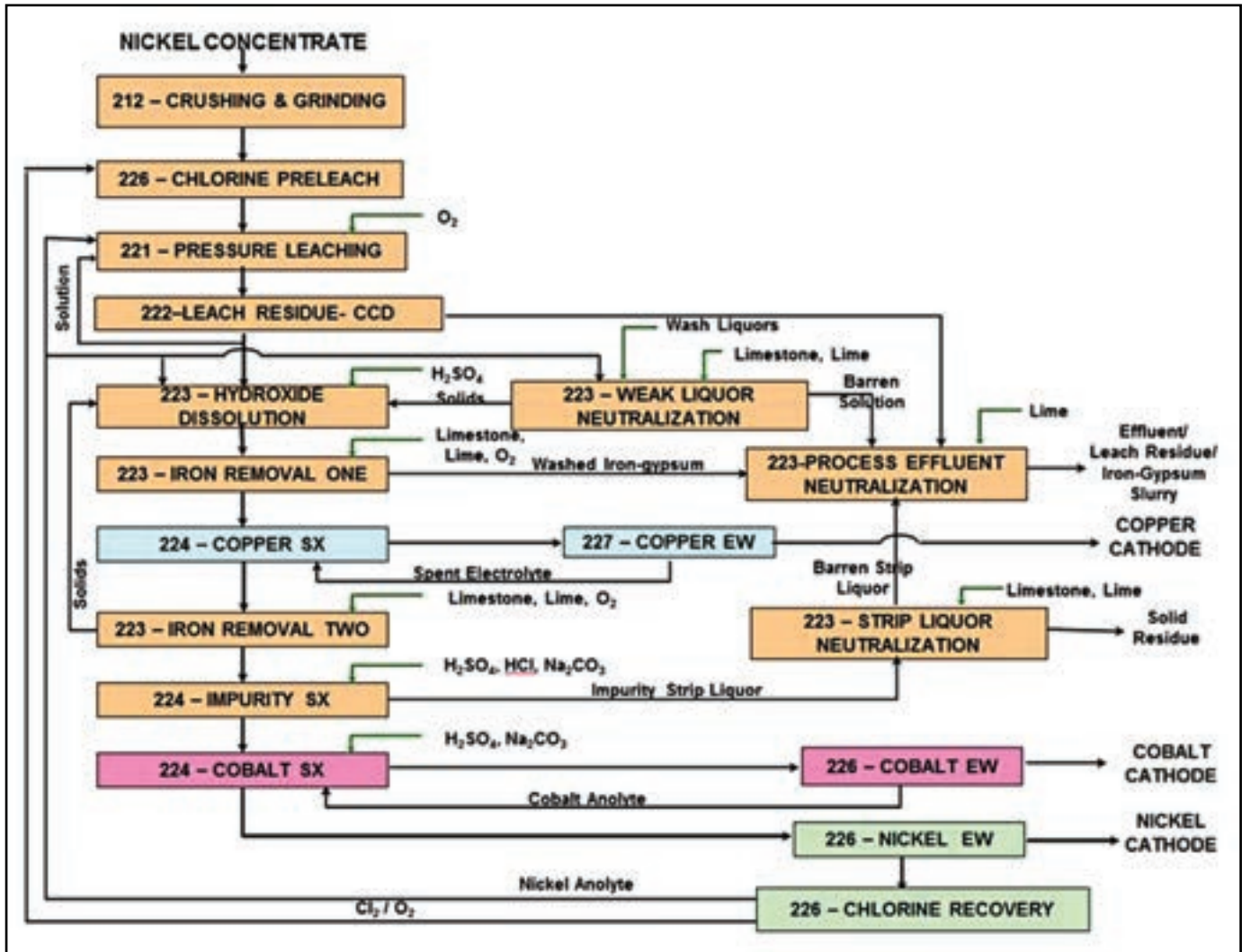
<sup>139</sup> Holloway et al. 2019, 1.

<sup>140</sup> Brent 2011, 24.

<sup>141</sup> Campbell 2015.



## Hydromet Pressure Oxidative Leach (POL) Process



**Figure 8:** Simplified block flow diagram of the hydromet process (Courtesy of Vale).

The hydromet process was chosen for the Voisey's bay project for several reasons. Like other hydrometallurgical processes, it provides extremely low aerial sulphur emissions, since the sulphur in the ore is removed as elemental sulphur in the tailings. Another advantage is scale. The capital investment required for smelting operations favour larger facilities than what was necessary for the Voisey's Bay deposit. The long-established Inco smelter and refinery in Sudbury has about twice the capacity of the Long Harbour facility.<sup>142</sup>

The chemical advantages of a chloride-enhanced pressure oxidative leach are discussed in further detail in the context of copper pressure leaching processes below. These benefits

include enhanced leaching rates, a low degree of sulphur oxidation, and the removal of iron as a solid residue.<sup>143</sup> The Voisey's Bay concentrate also favoured the hydrometallurgical route due to its lack of economically recoverable precious metals, which obviated the complicated and expensive process of recovering precious metals from the tailings of the chloride-assisted process.<sup>144</sup> Finally, the maritime location of the plant provided an acceptable environment for disposing of large quantities of primarily calcium effluent.

The primary feed material for the hydromet process is stored nickel-copper-cobalt concentrate that has been shipped to the Long Harbour site from the concentrator plant

<sup>142</sup> Brent 2011, 24.

<sup>143</sup> Love et al 2002.

<sup>144</sup> Goldie 2005, 127.

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at the Voisey's Bay mine. This is ground at a facility within the Long Harbour plant. The wet grinding step processes the concentrate, which begins with an average particle size of 100 microns, into a slurry consisting of 20-micron particles.

Following wet grinding, the slurry concentrate is pre-leached at atmospheric pressure with chlorine gas in a series of stirred tanks in order to activate the concentrate. The chlorine gas is harvested from the nickel electrowinning process. The main purpose of this pre-leach step is the efficient recycling of chlorine throughout the overall operation.<sup>145</sup>

The solids from this process undergo pressure leaching in an autoclave at high temperature with oxygen injection. The leaching agent is nickel anolyte recycled from the nickel electrowinning step. Nickel, copper, and cobalt are dissolved into a mixed sulphate and chloride solution. A small portion of the sulphide reacts with the injected oxygen to produce sulphuric acid, which further advances the leaching process. The majority of the iron in the concentrate is converted to insoluble ferric oxide, while the majority of the sulphide becomes molten elemental sulphur. Slurry is discharged through an external flash vessel where cooling occurs. The autoclave itself operates continuously and remains pressurized.<sup>146</sup>

The slurry then undergoes a separation step in which the leach liquor is divided from the residue in a thickener. The residue from this process is washed with water in a series of decantation thickeners to remove residual leach liquor. This acidic residue is then treated with lime to neutralize it, and blended with gypsum and iron hydroxide produced in the subsequent iron-removal step. The neutralized residue is piped to the tailings pond for long-term underwater storage (see below).<sup>147</sup>

The decanted leach solution from the thickener is contacted with nickel hydroxide, produced later in the process. This dissolves the nickel hydroxide while consuming most of the acid that the leach solution carries from the autoclave. Following the nickel hydroxide dissolution step, the solution proceeds to the first of two iron-removal steps. This involves neutralization with lime to remove silica, acid, and the majority of the iron.

The leach solution then proceeds to the copper solvent extraction step to remove copper. Copper is electrowon from the copper-rich solvent extraction strip solution to produce pure copper cathode. The copper-free solution then proceeds to a second iron-removal step in which it is treated with lime and oxygen to precipitate the remaining iron. The precipitate is recycled to the first iron-removal step to recover co-precipitated nickel.

Raffinate from the second iron-removal step enters the cadmium-removal circuit. As noted, this circuit was jointly developed with Sherritt Technologies and implemented at Long Harbour in 2017. This step reacts the raffinate with H<sub>2</sub>S gas in two of three pipe reactors (one is stand-by). The resulting slurry is filtered to remove precipitated cadmium. The process reduces the level of cadmium in the raffinate from around 1.4 milligrams per litre to less than 0.3 milligrams per litre.<sup>148</sup> The solution then proceeds to an impurity solvent extraction process to remove residual copper and iron, as well as lead, cadmium and other minor impurities.<sup>149</sup>

The solution, now consisting mostly of dissolved nickel and cobalt, proceeds to a cobalt solvent extraction where an extractant is used to remove cobalt. The strip solution proceeds to an electrowinning step, in which rounds of pure cobalt are produced. The solution then proceeds to a nickel electrowinning step to produce pure nickel rounds. Chlorine gas and oxygen given off by the electrowinning step are recycled to the pre-leach step. The majority of the anolyte is then recycled to the pressure leaching process. A smaller part is sent to the nickel hydroxide dissolution step in order to dissolve nickel hydroxide remaining in the anolyte.

A portion of the spent anolyte is sent to the weak liquor-neutralization step. This is a bleed stream meant to prevent the build-up of impurities that would occur through perpetual recycling of the anolyte. In this process, weak wash liquors from various steps are combined with the bleed-stream anolyte. The mixture is treated with lime to precipitate nickel hydroxide and other metals, which are then separated by thickening and recycled to the iron precipitation and nickel hydroxide dissolution step. The remaining barren solution is combined with overflow water from the tailings pond, treated, and then discharged into the ocean at Long Harbour bay.<sup>150</sup>

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<sup>145</sup> Mihaylov, Indje (March 20, 2020) Personal Phone Interview. 23:00.

<sup>146</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 7-3, 7-5.

<sup>147</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 7-3.

<sup>148</sup> Holloway et al. 2019, 4-5.

<sup>149</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 7-5; Crundwell et al. (2011), 154.

<sup>150</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 7-5. Crundwell et al. (2011), 154.

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## Water and environment

The remarkable scrutiny that modern capital projects undergo is evident in the provincial public records of the certification process for the Long Harbour Processing Plant. The Long Harbour environmental assessment, addressing both provincial and federal legislation, lasted from 2006 to 2008, with several further plans submitted following its approval.

The breadth of information required for approval of a project is extensive. It includes public health surveys of the surrounding communities, as well as the analysis of potential impacts on local flora and fauna. In this case, the company was required to identify “worst-case scenarios,” such as the failure of a tailings dam or release of chlorine gas; to illustrate development of equitable hiring policies; and to create detailed plans for eventual decommissioning.<sup>151</sup>

The final draft of the executive summary of the environmental impact statement (EIS), submitted by the Voisey’s Bay Nickel Company in April 2008, provides a succinct account of the project’s projected environmental footprint, from initial construction to decommissioning. It also provides an accessible introduction to the environmental and safety “best practices” associated with modern industrial projects in Canada. The following is a list of key overall environmental design principles cited for this project:

- Keeping Project boundaries as small as possible and minimizing watershed use;
- Reducing the size and extent of physical disturbance;
- Increasing the amount of recycling of resources such as water;
- Reducing the number of release points for the project (such as water discharges);
- Identifying environmentally sensitive areas and avoiding disturbance to these areas;
- Planning all aspects of the project for eventual closure.<sup>152</sup>

Of particular interest to this survey is the extensive documentation surrounding the use of water. Since hydrometallurgy requires a liquid medium, and releases its effluent in liquid form, the efficient use of water and the containment of effluent are prominent among environmental

considerations. The hydromet process uses around 4.4 million cubic metres of water per year.<sup>153</sup> In essence, the landscape surrounding the project becomes part of its broader economy of water use.

At the centre of this system is Rattling Brook Big Pond, which is the source of fresh water for the project. This water is treated with sodium carbonate in order to increase its alkalinity and reduce the risk of corrosion to the process machinery.<sup>154</sup> Blended with leach tailings into a liquid slurry, some of the water is piped to the Sandy Pond, which is used as a permanent tailings storage site. Water from Sandy Pond is recycled back into the plant or discharged to the ocean with the neutralized effluent stream.

The hydromet process produces a considerable amount of residue: 381,000 tonnes per year. Roughly 242,600 tonnes of this is neutralized leach residue containing about 25 percent elemental sulphur.<sup>155</sup> This sulphur requires isolation in order to prevent it from generating sulphuric acid in the presence of oxygen. Various options were considered based on trials of the residue from the test operations in Mississauga. Burial, whether as a paste or slurry, was found to “result in a long-term chronic acid-generating condition.” The acid produced over time would then leach and release high concentrations of metal from the residue. The resulting disposal site would have to be monitored and treated over a long period.<sup>156</sup>

After surveying a number of potential areas around the project, a candidate site was identified in Sandy Pond, a freshwater lake separated from the watershed and located 3.2 kilometres away from the site. The project also involved building three earth-filled dams to prevent contamination of other natural water sources.<sup>157</sup> Sandy Pond is connected to the hydromet plant with a 3.8-kilometre pipeline. This pipeline also returns clarified overflow water to the plant for process operations, and for neutralization and release to the marine environment.<sup>158</sup> Sandy Pond was chosen over an artificial containment pond because its floor has very low permeability. Extensive residue stability studies have shown low amounts of long-term leaching of metals into the surrounding environment.

The need for this freshwater tailings pond led to a further challenge with regards to environmental legislation. Sandy Pond was a natural fish habitat. The construction of the tailings pond entailed the destruction of that habitat. This was permitted under the federal Fisheries Act, which was

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<sup>151</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 2-8.

<sup>152</sup> Vale Inco Newfoundland & Labrador Limited 2008, Executive Summary, 6.

<sup>153</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 7-14.

<sup>154</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 7-14.

<sup>155</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 3-3.

<sup>156</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 3-4.

<sup>157</sup> Vale Inco Newfoundland & Labrador Limited 2008, Executive Summary, 11.

<sup>158</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 7-5.

amended in 2002 to include a “no net loss” principle. This allowed for the destruction of freshwater habitat for industrial development purposes provided that additional habitat was gained elsewhere.

In 2010, the planned use of the lake was challenged in court by a coalition of environmental and public policy groups called the “Sandy Pond Alliance to Protect Canadian Waters.” After a lengthy legal process, the coalition lost its challenge before the Supreme Court of Canada. However, this and other challenges to the use of freshwater lakes as tailings ponds contribute to a broader public debate about environmental policy and industrial development in Canada.<sup>159</sup>

While tailings from the plant are permanently stored underwater in Sandy Pond, a liquid effluent from the plant (distinct from the tailings just described) is released into the Atlantic Ocean. This is gathered in a separate storage tank from the residue. It is treated in a process-effluent neutralization clarifier, consisting of two neutralizing reactors, where it is blended with lime to raise its pH and precipitate dissolved metals.

The neutralization of acidic tailings from the process requires considerable infrastructure. Once neutralized, the effluent is held in a cooling and polishing pond, which is used to settle solids and further adjust the pH of liquid effluent. Finally, it is discharged through a pipeline leading to an underwater liquid diffusion system that opens into the mouth of Long Harbour at 50 to 70 metres of depth.<sup>160</sup>

A final significant aspect of the water-management scheme involves the various drainage control arrangements and other measures aimed at preventing freshwater contamination due to runoff. For instance, the flow of storm water through the facility is managed through a network of surface and subsurface drainage systems such as storm drains, ditches, and ponds. An oil-water separator is used to prevent the contamination of storm water flowing through the centralized fuel storage area.<sup>161</sup>

The environmental approval process notably includes a detailed plan for decommissioning the plant. This extends to the demolition and removal of buildings, foundations and other infrastructure. Other measures include the revegetation of the site, as well as the closure, stabilization, and long-term monitoring of the underwater residue storage.<sup>162</sup>

## THE UGS (UPGRADED SLAG) PROCESS FOR TITANIUM DIOXIDE PRODUCTION

Titanium is the ninth most abundant element in the earth’s crust. It is economically recoverable when it occurs in concentrated natural deposits, either as rock or sand. The vast majority of the world’s titanium production comes in the form of titanium dioxide rather than titanium metal. Titanium dioxide is an excellent white pigment that is both bright and chemically stable. It is used primarily in the pigment industry as a colorant and an opacifier, though it has many other uses. Since production first began in the early decades of the twentieth century, titanium dioxide pigment has become an essential part of the built landscape, though most of us are ignorant of its presence.<sup>163</sup>

Since the 1950s, Quebec has been a major producer of the precursor materials used to manufacture this pigment. This industry was founded on the massive rock ilmenite deposits in the Allard Lake region most notably the Lac Tio deposit, which is among the largest rock ilmenite deposits in the world. As is normally the case, titanium dioxide in this deposit is associated with iron.<sup>164</sup> Typical (“run-of-mine”) hemo-ilmenite ore from Lac Tio consists of about 32.7 percent titanium oxides and 37 percent iron oxides by mass, with additional minerals such as silicates (6.65 percent), aluminum and magnesium oxides (4.14 percent and 2.92 percent, respectively), and other gangue materials.<sup>165</sup>

Discovered in 1946, the Lac Tio deposit is located 43 kilometres north of the Port of Havre-Saint-Pierre on the St. Lawrence River. Following rail transportation to the port, the ore is shipped roughly 885 kilometres to the industrial city of Sorel-Tracy, which sits at the confluence of the Richelieu and St. Lawrence rivers in the Montérégie region of Southern Quebec. Here, the ore is processed at the Sorel-Tracy Metallurgical Complex, which currently operates nine electric furnaces producing iron and titaniferous slag marketed as SORELSLAG. Both of these products are upgraded at the facility.

For decades, the SORELSLAG product from this process was successfully sold as a precursor material to the pigment industry. However, advances in pigment production, along with competition from abroad, motivated researchers at

<sup>159</sup> Ballam 2013.

<sup>160</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 4-1.

<sup>161</sup> Vale Inco Newfoundland & Labrador Limited 2008, Executive Summary, 19, 28.

<sup>162</sup> Vale Inco Newfoundland & Labrador Limited 2008, Volume 1, 8.0-8.1.

<sup>163</sup> Filippou and Houdon 2009, 36.

<sup>164</sup> Filippou and Houdon 2009, 36.

<sup>165</sup> Gueguin and Cardarelli 2007, 17.

QIT-Fer et Titane (now Rio Tinto Fer et Titane, or RTFT) to develop a more refined product. Since 1997, a new process, known as UGS, or upgraded slag, produced a refined product that has sold successfully to pigment manufacturers. Substantial upgrades to the capacity of that facility, as well as the overall plant, testify to the success of this novel hydrometallurgical process.

## History and development of titanium dioxide production at Sorel-Tracy.

The expansion of heavy industry in Quebec in the period surrounding the Second World War forms the background to the development of the Sorel-Tracy facility. At that time, the hydroelectric industry, which had developed since the early decades of the twentieth century, underwent a significant expansion to meet the demands of the Allied war effort.

Many industries, including aluminum smelting and various forms of chemical manufacturing, are extremely energy-intensive. As such, their facilities have historically emerged in clusters around centres of energy production. Quebec's developing hydroelectric industry provided a catalyst for the establishment of heavy industry around the Great Lakes-St. Lawrence River Basin over the first half of the early twentieth century. The smelting of titanium ore from Quebec's rich ilmenite deposits is one industrial activity among many to have profited from these advantages.

As with Quebec's aluminum smelting industry, the critical factor in titanium dioxide production was access to the energy necessary to run electric arc furnaces. In 1951, as the Sorel-Tracy facility was being developed, *Macleans*' magazine noted: "Power in Quebec is so cheap that authorities can afford under some circumstances to offer big lumps of it to industries for free."<sup>166</sup> Both the Quebec-based aluminum industry (formerly Alcan) and its titanium dioxide production have since been purchased by the Rio Tinto multinational, which now controls the former Alcan hydroelectric infrastructure.<sup>167</sup>

In 1941, the Quebec Geologic Survey discovered significant deposits of rock ilmenite in the Allard Lake region of Quebec. A number of claims were staked following the publication of the provincial government report on the find. The discovery arrived at a welcome time. North American production of titanium dioxide had previously relied on ore from India, the supply of which had been disrupted by the Second World War. This created a dependence on lower-grade North American ore, and a concerted search for better deposits.<sup>168</sup>

The Quebec claims were purchased by Canadian subsidiaries of the Kennecott Copper Corporation and the New Jersey Zinc Company. In 1946, the area was further explored by Kenneco Exploration, which identified a number of promising ilmenite deposits. By 1948, plans were made to bring the deposit into production.<sup>169</sup> That year, construction began on a railway to transport the ore from the Lac Tio mine site to the fishing port of Havre-Saint-Pierre. The ore would then be shipped to Sorel, Quebec, for processing.

The new operation, called the Quebec Iron and Titanium Corporation, was jointly owned by Kennecott Copper and the New Jersey Zinc.<sup>170</sup> Until 2010, the company would be known as QIT Fer et Titane (popularly as "La QIT" or "Québec Iron"). At that point, its name was formally changed to Rio Tinto to reflect the ownership of the Anglo-Australian multinational that had, by that point, owned the company for 20 years. Company sources refer to the operation as Rio Tinto Fer et Titane, or RTFT.<sup>171</sup>

The first electric furnace of the Sorel smelter complex was constructed between 1949 and 1951. Several more were added in subsequent years. In 1949, researchers with QIT Fer et Titane, registered in New York, patented a process for creating a titanium slag concentrate that, upon leaching, produced a leach solution with approximately 80 percent titanium.<sup>172</sup> This product, sold as "SORELSLAG," is produced by smelting the ilmenite ore with anthracite coal in an electric arc furnace. Initial annual output of titanium slag for 1950, the first year of operation, was 250,000 tonnes.<sup>173</sup>

This product was destined for the production of titanium dioxide pigment via the sulphate process. This involves dissolving a titania-rich feedstock into sulphuric acid before precipitation, washing, drying, and calcining it into the pigment product.<sup>174</sup> Commercial production of this white pigment began in Norway in 1916, and in the United States two years later. Following the development of titanium oxide-based paints in 1921, production expanded significantly, with new plants opened across the United States. Over this period, the pigment became ubiquitous as it replaced basic lead carbonate, the previous standard white pigment that has become known for its toxicity.

As its name implies, QIT-Fer et Titane also produces iron in its electric arc furnaces. This was initially sold as pig iron, but the company has, over the years, produced an increasing range of iron products. In 1968, for instance, the company added an iron powder plant to produce a product

<sup>169</sup> Hammond 635

<sup>170</sup> Hammond 635

<sup>171</sup> Doyon 2010.

<sup>172</sup> Peirce et al. 1949.

<sup>173</sup> Guéguin and Cardarelli, 2.

<sup>174</sup> Guéguin and Cardarelli, 38.

<sup>166</sup> Bodsworth 1951, 47.

<sup>167</sup> St-Hilaire 2012.

<sup>168</sup> Hatch and Cuke 1956, 619.

marketed as “SORELMETAL.” Steelmaking and steel powder production were added, with additional plants, in the mid-1980s.<sup>175</sup> The steelmaking plant was the final major investment before the development and implementation of the UGS process.

The UGS process was designed to address a decline in the commercial prospects of SORELSLAG. The primary reason for this was the increasing popularity of the chloride process for titanium dioxide production. Piloted by DuPont in the United States in 1948, and further developed over the 1960s, the chloride process is considered to produce a better pigment than the sulphate method. It also has environmental advantages, and produces titanium tetrachloride as an intermediate product. This can be sold as a raw material in the production of titanium metal.<sup>176</sup> The current chloride process involves vaporizing feedstock in a stream of chlorine gas before selectively condensing it to remove impurities, and subsequently converting it to pure TiO<sub>2</sub> through contact with oxygen at high temperatures. The chloride process has steadily gained market share over the sulphate process, though ever-increasing Chinese production has largely relied on the sulphate process.

Whereas the sulphate process simply requires that the feedstock be soluble in sulphuric acid, the chloride process has several additional requirements. For instance, chloride feed requires a low level of alkaline earth impurities, the particles must be within a particular size range, and the proportion of titanium dioxide must be as high as possible.<sup>177</sup> In all of these respects, SORELSLAG was incompatible with the chloride process. In addition, by the 1990s, the market for SORELSLAG had declined significantly due to competition from producers in the former Soviet Union.<sup>178</sup>

Faced with these challenges to the operation’s future viability, the company dedicated their resources to developing a new process for upgrading its titaniferous slag for compatibility with the chloride process. The company had founded a technology centre in 1967 to optimize plant processes and engage in strategic R&D. Over subsequent years, using state-of-the-art equipment, the centre had conducted analyses of the company’s various ores and products.<sup>179</sup>

Work began on what would become the UGS process in the early 1990s. In November 1995, a U.S. patent was filed by the key inventors of the process: Krzysztof Borowiec, Alfonso

E. Grau, Michel Guéguin, and Jean-François Turgeon. The patent was granted in November 1998.<sup>180</sup> This patent followed much previous work by researcher Michel Guéguin to produce material suitable for use in the chloride process. This earlier work was dedicated to upgrading ilmenite ore to a synthetic form of rutile, a mineral rich in titanium dioxide. Following two years of process development and optimization, construction of the new plant began on January 30, 1996. At a total cost of CAN\$430 million, this was the largest construction project in Quebec while it was underway. The massive undertaking included the rebuilding of four of the nine electrical furnaces at the plant, in order to increase their capacity. The first upgraded slag product was produced in December 1997. The process produces a titanium dioxide product with a purity of 94.5 percent.

The success of this process is evident in subsequent investments made to expand its capacity in order to meet growing demand. Between 2004 and 2006, the UGS plant underwent a major expansion, costing several hundred million dollars, that increased the plant’s output by 15 percent. Additional production lines were added to the various constituent plants, while the overall process was optimized. In 2018, a further CAN\$43 million was invested to refurbish an electric furnace that had been shuttered in 2013 due to a downturn in the market.<sup>181</sup>

The UGS process has also permitted the Sorel-Tracy Metallurgical Complex to flourish as a processing plant for ilmenite from around the world. The plant notably processes ilmenite sand mined by Rio Tinto’s QIT Madagascar Minerals (QMM) operation at the Fort Dauphin deposit in Madagascar. Preparation for processing of this new feedstock required the addition of US\$195 million in new investment. The first shipment of Madagascar concentrate arrived on June 11, 2009. This concentrate is transformed into Rio Tinto chloride slag (RTCS), which can be used directly in chloride pigment production, and is not upgraded using the UGS process.<sup>182</sup>

In 2018, the Sorel-Tracy metallurgical facility produced 1.3 million tonnes of titaniferous slag and 1 million tonnes of cast iron. Since its 2006 expansion, the UGS plant boasts a production capacity of more than 375,000 tonnes per year. Meanwhile, as of 2019, the RTFT operation had an estimated 149 million tonnes of ore reserves.<sup>183</sup>

<sup>175</sup> Brown et al. 83.

<sup>176</sup> Filippou and Houdon 2009, 36.

<sup>177</sup> Borowiec et al. 1998.

<sup>178</sup> Rio Tinto (2017).

<sup>179</sup> Rio Tinto (2017).

<sup>180</sup> Borowiec et al.1998.

<sup>181</sup> Rio Tinto 2018.

<sup>182</sup> Rio Tinto 2009, 45.

<sup>183</sup> Rio Tinto 2017.

## SORELSLAG and the UGS process

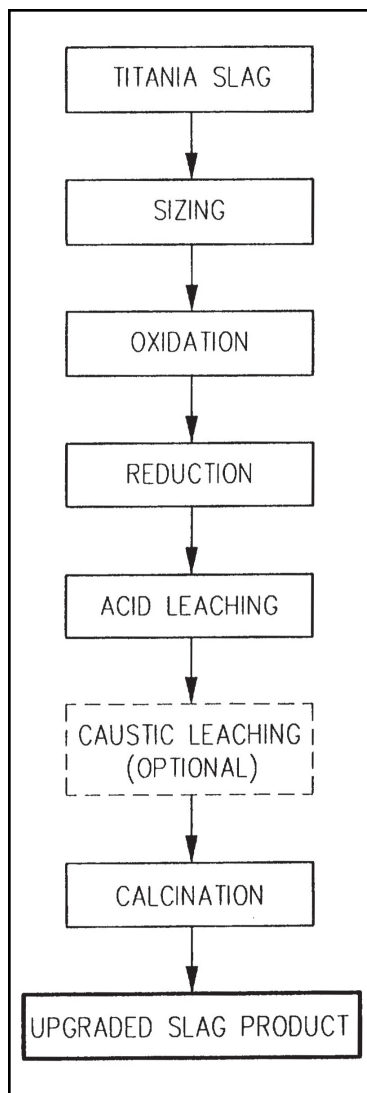
Before being shipped to the Sorel-Tracy complex and entering the UGS process, ore from the Lac Tio deposit (32.7 percent TiO<sub>2</sub> by weight) undergoes an initial beneficiation process: it is crushed at the mine site in order to concentrate the ore and remove gangue minerals. Once at Sorel-Tracy, it undergoes a secondary crushing, followed by screening, gravity beneficiation, and dewatering. The beneficiated ore (34.5 percent TiO<sub>2</sub>) is then roasted in air heated to a temperature ranging from 900 to 1000°C. This oxidizing process permits the removal of sulphur and creates ferromagnetic domains within the hemo-ilmenite particles, increasing their magnetic susceptibility. These particles are then separated before using dry magnetic separation. This “upgraded roasted ore” (URO) contains 37.8 percent titanium dioxide.<sup>184</sup>

This URO material is then smelted with anthracite coal in electric arc furnaces, a process known as electrothermal reduction. The power of these furnaces ranges from 50 to 70 MW.<sup>185</sup> The heavier molten iron-carbon alloy separates from a titania-rich lighter slag and is tapped from the bottom of the furnace. A portion of this iron is cast and sold as “high-purity pig iron” HPPI. The remainder is purified of residual sulphur and processed into various products, including powders and steel, at an iron and steelmaking plant.<sup>186</sup>

The titanium-rich slag, containing around 80 percent titanium dioxide, is tapped into lined steel wagons. Once the slag has cooled sufficiently to form a solid crust or “skull,” it is demoulded and left to air-cool until the inner core has solidified. This is then crushed, ground, and screened to a consistent size at a sizing and preparation plant to create SORELSLAG.<sup>187</sup>

While SORELSLAG continues to be sold as a feed material for sulphide pigment production, a portion is upgraded into the UGS product. The UGS process begins in the sizing and preparation plant, where particles are processed by grinding, screening, and classifying, to produce particles at a diameter suitable for the chlorine process—ideally between 250 and 350 microns.

The slag then undergoes a high-temperature pre-treatment in an oxidation-reduction plant (ORP). The oxidization process takes place around 1000°C, rendering the titanium to an insoluble state. This also decomposes glassy silicates containing silicone and calcium oxides, which facilitates their dissolution in the subsequent leaching phase.<sup>188</sup> The reduction phase, which takes place at 800°C,



**Figure 9:** Simplified flowchart from the patent of the UGS process (adapted from Borowiec et al. 1998). For a more detailed diagram of the process as implemented, see Gueguin and Cardarelli 2007, 39.

reduces iron oxide to ferrous oxides. The product of this operation is called “heat-treated slag.”<sup>189</sup>

This material is then processed in the acid-leaching plant where it is treated in an autoclave at elevated temperature and pressure with hydrochloric acid used as the leaching liquid. The leaching takes place in two stages, with new acid added at each stage. Leaching usually takes from five to seven hours. This process dissolves impurities into the leach solution. The solids are then washed and calcined at 600 to 800°C to produce the final UGS product.<sup>190</sup> Spent acid is treated in the acid regeneration plant, which employs a pyro-hydrolyser that combines spent acid with hot gases. Dissolved impurities in the acid are deposited on particles in an existing bed of oxides, while hydrogen chloride gas yields hydrochloric acid that is returned to the leaching circuit.

<sup>184</sup> Gueguin and Cardarelli 2007, 22-23.

<sup>185</sup> Filippou and Houdon 2009, 37.

<sup>186</sup> Filippou and Houdon 2009, 37.

<sup>187</sup> Gueguin and Cardarelli 2007, 27-29.

<sup>188</sup> Borowiec et al. 2002.

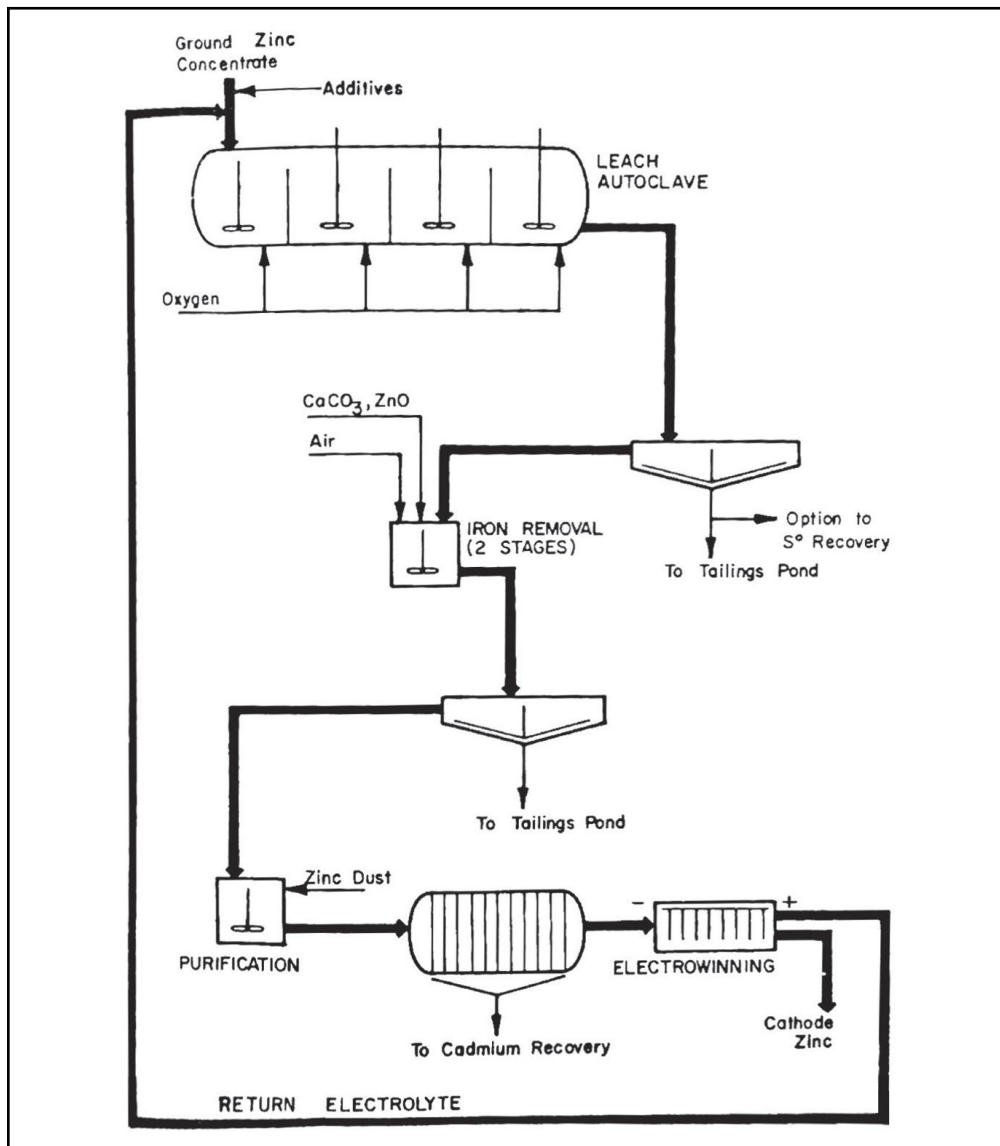
<sup>189</sup> Gueguin and Cardarelli 2007, 42.

<sup>190</sup> Collins et al. 2011, 306-307.

# SHERRITT'S ZINC PRESSURE LEACH PROCESS

Zinc is an economically important base metal with key applications in battery production and as a protective coating for iron alloys. The Sherritt zinc pressure leach process was the company's first pressure hydrometallurgical process to be successfully applied beyond Fort Saskatchewan. Since 1981, when the world's first zinc pressure leaching plant was commissioned at Trail, British Columbia, the process has proven to be among the more successful applications of pressure hydrometallurgy.

The conventional method of processing zinc is a hybrid roast-leach-electrowin operation. Sherritt's zinc process was applied as a supplement to the initial roasting stage of the conventional process. In most of its implementations, it was not a replacement for existing zinc production facilities, but rather a means to expand production without a corresponding increase in sulphur dioxide emissions and sulphuric acid production.<sup>191</sup> This changed with the implementation of a two-stage leach plant opened at HBMS (Hudson Bay Mining and Smelting Company, now Hudbay Minerals) in 1993, which entirely replaced the existing operation. The process was also designed to produce elemental sulphur and to obtain an initial zinc-extraction rate of above 95 percent.<sup>192</sup>



**Figure 10:** Single Stage Zinc Pressure Leaching (Parker and Romanchuck 1980, 423).

<sup>191</sup> Ozberk et al 1995, 49.

<sup>192</sup> Collins et al, 1994, 58.



## HISTORY AND DEVELOPMENT OF ZINC PRESSURE LEACH

The Sherritt zinc pressure leach process operates basically as follows: A finely ground zinc concentrate is processed with sulphuric acid and injected oxygen in a pressurized multi-compartment autoclave at around 150°C. The sulphuric acid leach is spent electrolyte returned from the electrolysis cells, where the acid is generated during the refining process. About 95 to 96 percent of sulphide sulphur is converted to elemental sulphur during this process.<sup>193</sup> Dissolved iron in the leach increases the rate of oxidation. Once the reaction has consumed a sufficient amount of free acid, iron precipitates from the solution as basic iron sulphate and jarosite.<sup>194</sup>

A zinc sulphate solution—a zinc-enriched slurry—is filtered from the leach residue. This solution then requires further processing in order to remove impurities such as iron and lead, and, in some cases, to recover precious metals. When used as an addition to an existing zinc plant, the process of purifying the leach solution is accomplished using the “neutral leach” system that is already present. Neutral leach incorporates calcine from the roasting facilities to neutralize the acid leach solution before iron removal.

In the case of stand-alone, or “grassroots,” leaching plants, such as the two-stage system implemented at the Hudbay Minerals refinery in Flin Flon, a different method was developed to remove iron and to purify the solution before electrowinning.<sup>195</sup> This involves a two-stage counter current system using acid generated in the electrowinning stage. The first, “neutral leach” stage, uses the spent acid from the second stage, which is neutralized using fresh concentrate.

In either case, the leach residue is filtered to separate elemental sulphur from a sulphide residue. The sulphur may then be stockpiled, sold, or burned to produce acid. The residue may be reprocessed depending on the nature of the concentrate. For instance, when the two-stage Hudbay plant was put into operation, the treatment of sulphur residue depended on the gold content of the feed. The sulphur was either impounded with the pressure leach tailings, or captured through flotation and hot filtration, to permit the reprocessing of gold-containing residual sulphides.<sup>196</sup> In the case of the Cominco operation described below, a zinc-enriched sulphide cake is sent to the roasting section. The neutralized leach solution continues to an iron-removal process and is purified before arriving at the electrowinning stage.

In the late 1950s, the Sherritt research group that had developed the Sherritt ammoniacal process for nickel began work on pressure leaching zinc. This group included early pioneers of Canadian metallurgy such as Frank Forward and Vladimir Mackiw.<sup>197</sup> The first successful production of zinc solution and elemental sulphur took place in 1959.<sup>198</sup> The process was initially limited to relatively low temperatures, below the melting point of sulphur (119°C), in order to prevent molten sulphur from limiting extraction by coating unreacted sulphides. This resulted in lengthy, six- to eight-hour leaching times.

By the late 1970s, researchers had discovered chemical dispersants that would effectively prevent the occlusion of unleached material by molten sulphur, making the process much more viable.<sup>199</sup> This permitted the process to operate more quickly and at higher temperatures. It also allowed for the use of a smaller autoclave, reducing capital costs. Likewise, development work by Cominco had produced an efficient means of separating molten sulphur from the leach solution.<sup>200</sup>

With interest in the process growing, Sherritt undertook large-scale piloting, with the intention of commercializing the process. An ongoing collaboration with Cominco on the Sherritt-Cominco copper process (discussed in the following chapter) provided the basis for joint work on this technology. Cominco’s existing zinc production offered a logical place to test the process on a commercial scale.<sup>201</sup>

Sherritt first analysed the possibility of applying a one-stage pressure leach process to lead-zinc ores in 1977, using a lead-zinc concentrate from New Brunswick. Further work in this area was carried out in the 1980s, under a funding agreement between Sherritt and CANMET, to improve the grade of the residue-bearing lead and silver.<sup>202</sup>

<sup>193</sup> Collins et al 1990, 299.

<sup>194</sup> Collins et al, 1994, 52.

<sup>195</sup> In 2004, the Hudson Bay Mining and Smelting Company Limited (HBMS) was renamed Hudbay Minerals Incorporated. Except in the case of historical circumstances, this report will refer to the company as Hudbay.

<sup>196</sup> Krysa 1995, 75.

<sup>197</sup> See: Parker and Romanchuck 1980, 407 ; Ozbert et al 1995, 50-51.

<sup>198</sup> See: Forward, F. A. and Veltman, H. “Direct Leaching Zinc Sulphide Concentrates by Sherritt Gordon,” *Journal of Metals* 12 (1959), 836-840.

<sup>199</sup> For a brief account of the development of chemical dispersants in the context of zinc hydrometallurgy see: John Dutrizac, interviewed by William McRae, February 2016, Mining and Metallurgy Legacy Project. <https://www.youtube.com/watch?v=GNENgEBQWqQ> (accessed March 18, 2019), 15:00.

<sup>200</sup> Parker and Romanchuck 1980, 408.

<sup>201</sup> Collins, Michael. (December 7, 2018) Personal Phone Interview. 18:00.

<sup>202</sup> Chalkey et al 1993, 938.

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# THE INTRODUCTION OF SINGLE-STAGE ZINC PRESSURE LEACH AT COMINCO

The first full-scale application of the process was at the Cominco (now Teck Resources) Trail plant in British Columbia in 1981. First established in 1896, the metallurgical complex at Trail had evolved from a copper-gold smelter to a flexible processing centre for ores from around the B.C. interior. A series of upgrades had garnered the facility an international reputation as a sophisticated, integrated complex focussing on lead and zinc processing—but also as a major regional polluter.<sup>203</sup> By the mid-1970s, it became clear that increased production must go hand-in-hand with emission reduction: this would require investment in a series of new plants.

Cominco had first shown interest in Sherritt's development work on the zinc pressure leach process in the early 1960s. In 1962, the two companies collaborated on a small test leaching autoclave using zinc concentrate from Sherritt's Sullivan mine in Kimberly, B.C. Though results were promising, the process was not implemented.<sup>204</sup>

In 1977, after surveying existing technologies, Cominco committed CAN\$425 million to modernize both the Trail plant and the Sullivan mine.<sup>205</sup> Under this plan, zinc production would increase from 227,000 to 272,000 tonnes per year. The company envisioned the installation of a new zinc pressure leach plant that would process around 25 percent of the zinc concentrate, and feed into the neutral leach operation. This would be the first plant of its kind in the world.

In the summer and fall of 1977, the two companies operated a three-tonne per day pilot plant at Sherritt's research facility in Fort Saskatchewan, Alberta. A six-week commissioning process began in July of that year, followed

by an eight-week test and demonstration period. Operating on Cominco's Sullivan concentrate, the plant demonstrated both the pressure leaching process and the filtration of elemental sulphur.<sup>206</sup>

The new plant began operation in 1980, with a single autoclave treating 64,000 tonnes of zinc concentrate, and producing 31,000 tonnes of zinc metal per year.<sup>207</sup> Over subsequent years, numerous changes and improvements were introduced to solve emerging problems and to introduce efficiencies. In early 1982, for instance, the autoclave used to separate elemental sulphur failed. This required the development of an alternate approach involving a hydrocyclone, which was installed the following year.

In 1988, a major renovation costing CAN\$2.8 million was undertaken to improve several aspects of the pressure leach plant. Together, these changes resulted in major production improvements. That year, the plant operated at 193 percent of its original design capacity.<sup>208</sup> In 1996, the autoclave used for zinc pressure leaching was replaced with a vessel twice the size. This was to accommodate concentrate from Cominco's Red Dog mine in Alaska, which required longer leaching times.<sup>209</sup> A hot acid leaching step was also added to improve zinc recoveries in the initial leach.

The successful implementation of zinc pressure leach at Trail was followed in 1983 by a plant in the Kidd Creek Mines (now Falconbridge, Kidd Creek Division) located in Timmins, Ontario. In 1991, the technology was licensed to Ruhr-Zink GmbH in Datteln, Germany. These were also single-stage additions to existing roast-leach-electrowin operations.

The most ambitious and complex implementation of the Sherritt zinc pressure leach process was the plant commissioned in 1993 at the Flin Flon operation of Hudbay. This was the world's first two-stage pressure leach operation for zinc.<sup>210</sup> It was unlike the earlier three plants, in that it was a two-stage operation designed to completely replace the facility's polluting and out-dated multi-hearth roasters.

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<sup>203</sup> See Weidenhammer 2018, 35-41.

<sup>204</sup> Parker and Romanchuck 1980, 408.

<sup>205</sup> Fish 1980, 49-50.; Ashman and Jankola 1990, 254.

<sup>206</sup> Parker and Romanchuck 1980, 407-498.

<sup>207</sup> Parker and Romanchuck 1980, 421.

<sup>208</sup> Ashman and Jankola 1990, 254-257, 269.

<sup>209</sup> Ashman et al. 2011, 153.

<sup>210</sup> Krysa 1995, 71.

## IMPLEMENTING TWO-STAGE ZINC PRESSURE LEACHING AT FLIN FLON

Zinc operations began at the HBMS refinery in Flin Flon, Manitoba, in 1930. By the late 1960s, the need to renovate the obsolete and polluting roast-leach-electrowin plant became clear. The plant's isolation meant that any attempt to capture sulphur dioxide emissions for conversion to sulphuric acid would have incurred significant shipping and storage costs. These circumstances led to a search for hydrometallurgical processes that could be applied to the plant's operations. Options considered for replacing the plant's copper operations are discussed in the following chapter.

The two-stage zinc operation intended for the HBMS refinery was first tested in 1984 at the Sherritt Research Centre using a miniplant with a capacity of five kilograms per hour of concentrate. This trial provided the basis for the design of a much larger facility. The decision to go ahead with the plant's construction was taken in 1990.



**Figure 11:** A view of the zinc pressure leach autoclaves and piping systems at Hudbay's Flin Flon plant (Gil Desrochers Collection HBMS EPSN 2036, Courtesy of the Flin Flon Heritage Project).

The engineering design phase began in the spring of the following year.<sup>211</sup>

While this was a complex and novel process, the facility encountered relatively few problems.<sup>212</sup> In fact, by 1995, the process had exceeded its design specifications for zinc production, while eliminating sulphur dioxide and particulate emissions from the zinc facility.<sup>213</sup> This resulted in a 25 percent reduction in overall plant emissions, or about 89 kilotonnes per year less sulphur dioxide.<sup>214</sup> Since its commissioning, the facility has seen incremental improvement and expansion. For instance, in 2001, the addition of a new tank house for electrolytic refining permitted an increase to 124,000 tonnes per year of cast zinc.<sup>215</sup> In 2017, the Flin Flon plant produced 107,946 tonnes of cast zinc.

In the decades since the stand-alone zinc plant was developed at Flin Flon, the Sherritt zinc pressure leach process has been implemented at several other plants around the world. In 2003, a similar stand-alone plant, developed through experience gained from the Hudbay operation, was opened at Kazakhmys Balkhas' facilities in Kazakhstan. The plant ended operation in 2008, due mainly to a shortage of concentrate.<sup>216</sup>

In 2009, another two-stage operation was opened for Shenzhen Zhongjin Lingnan Nonfermet, located in Shaoguang in the Guangdong province of China. This operation also recovers a lead-silver byproduct, along with gallium and germanium. Finally, a two-stage plant processing bulk concentrates was opened by the China Western Mining Company in 2015. This plant is notable for its high-altitude location, at 2,600 metres above sea level. The zinc pressure leach process is currently used at four facilities: Trail, Flin Flon, and the two plants in China.<sup>217</sup>

<sup>211</sup> Collins et al, 1994.

<sup>212</sup> Krysa 1995, 75-76.

<sup>213</sup> Qiu and Krysa 2008, 180.

<sup>214</sup> Collins et al, 1994, 58.

<sup>215</sup> Qiu and Krysa 2008, 180-181.

<sup>216</sup> Svens 2012, 194-195.

<sup>217</sup> Svens 2012, 195-19.

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# PRESSURE LEACHING COPPER

A great deal of effort has been invested in developing a fully hydrometallurgical method for processing chalcopyrite (copper sulphide). Compared with other domains, these efforts have met with comparatively little success. While leaching and solvent extraction have been widely applied to low-grade sulphide and oxide ores, no process has been adopted for the more abundant chalcopyrite ores that feed most of the world's copper production.<sup>218</sup> In 2007, Canadian metallurgist and historian Fathi Habashi reflected on this history in a paper entitled “Abandoned but Not Forgotten—The Recent History of Copper Hydrometallurgy.” Within, Habashi lists eight processes, developed between 1970 and 1995, that have since been definitively abandoned.<sup>219</sup>

The main reason for this meagre result seems to be the increasing efficiency of the traditional smelting process. Pyrometallurgical developments such as the oxygen flash furnace have made the smelting process more effective and less polluting by producing concentrated exhaust streams that can be captured to sulphuric acid.<sup>220</sup> Smelting also has the inherent advantage gaining energy from combusting sulphur. Heat from combustion can be recycled, introducing further efficiencies.

The conventional hydrometallurgical processing of copper concentrates involves roasting of crushed ore or flotation concentrate, followed by leaching. Metallic copper is produced by electrowinning the purified leach solution. Where heap leaching is practicable, sulphuric acid from the roasting process is a useful leaching agent rather than an inconvenient byproduct. The usefulness of sulphuric acid may have been a factor preventing the wider adoption of a purely hydrometallurgical process.

Canadian researchers have made several contributions to this area of hydrometallurgy. These efforts point to the particular circumstances of Canadian copper producers. In 1978, for instance, researchers at Sherritt and Cominco noted that between 1.5 and 6 tonnes of sulphuric acid was generated for each tonne of copper produced by smelting, with the average being 4.5 tonnes.<sup>221</sup>

As with other non-ferrous metals, the advantages sought by eliminating the roasting step at remote Canadian copper

smelters such as Trail, B. C. and Flin Flon, Manitoba lie in the ability to produce elemental sulphur rather than sulphuric acid. Canadian ores are less amenable to the heap leaching and solvent extraction techniques now used for many low-grade chalcocite ores, while Canada's climate limits the viability of heap leaching.<sup>222</sup>

Today, it seems the prospects for the widespread adoption of hydrometallurgical processes for chalcopyrite are not promising. While such processes may find particular applications to challenging concentrates, when applied to conventional ores, they currently do not scale as well as smelting. Claims regarding the capacity to process “dirty” concentrates containing arsenic should be balanced against the challenge of coping with contaminated tailings.<sup>223</sup>

A range of hydrometallurgical leaching processes have been explored for chalcopyrite, including several approaches involving bioleaching—the use of living organisms to selectively remove targeted metals.<sup>224</sup> The Canadian-developed pressure leaching processes discussed below fall into two categories: they are either entirely acid-based, or they incorporate chloride to one extent or another. Both routes have particular advantages and disadvantages.

Chloride-based leaches tend to have relatively higher reaction rates, though the reagents are more expensive and much more corrosive. As a result, the associated plants tend to be costly and challenging to build. For instance, the need to include exotic materials, such as titanium autoclaves, in order to commission the CESL/Vale copper test plant in the Carajás region of Brazil, delayed that project considerably.<sup>225</sup> Acid-based leaches are cheaper to operate, since the reagents are more easily generated from the sulphide feed. These plants are cheaper to build using conventional materials, though the leaching times are longer.

The earliest of the processes discussed below, the sulphuric acid-based Sherritt-Cominco process for chalcopyrite, represents an implementation of Sherritt's pressure leaching legacy following the success of the ammoniacal leaching process. It also forms the backdrop for the collaboration between the two companies on the successful launch of the zinc pressure oxidization process at the Trail refinery. Lastly, it illustrates some of the challenges faced in the early development of sulphuric acid pressure leach, before the development of effective dispersant chemicals permitted the leach to operate at higher temperatures, and the development of solvents greatly simplified the purification of the leach liquor prior to electrowinning.

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<sup>218</sup> Ramachandran, Lakshmanan, and Kondos 2007, 101-102.

<sup>219</sup> Habashi 2007, 305.

<sup>220</sup> Weidenhammer 2018, 88-90; Ramachandran, Lakshmanan, and Kondos 2007, 102-103.

<sup>221</sup> Maschmeyer, Milner, and Parekh 1978, 133.

<sup>222</sup> Deschênes et al. 2011, 206.

<sup>223</sup> See Ramachandran, Lakshmanan, and Kondos 2007, 116-119.

<sup>224</sup> See Ramachandran, Lakshmanan, and Kondos 2007 for a thorough review of recent hydrometallurgical processes for chalcopyrite.

<sup>225</sup> See Defreyne and Cabral, 2009.

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## EARLY STEPS: THE SHERRITT-COMINCO COPPER PROCESS

Beginning in the 1970s, two major Canadian mining companies, Sherritt and Cominco, collaborated on a novel hydrometallurgical process for producing copper. Both companies controlled copper ore bodies and shared an interest in developing a process that produced sulphur rather than sulphuric acid and could effectively recover other constituent metals such as molybdenum, zinc, and precious metals.

Both required a versatile process that could be applied to a range of ores—a factor that likely led to the complexity of the final process. For instance, Cominco had a large supply of bornitic ores available from its stake in the Highland Valley copper deposit near Logan Lake, British Columbia.<sup>226</sup> The industry as a whole faced tightening regulations around the production of sulphur dioxide emissions. Both companies were interested in licensing the technology should it prove successful.<sup>227</sup>

Development of the Sherritt-Cominco process was abandoned before it could be commercially implemented. As a result, most information comes through three articles, published in 1978, that detail the piloting process and the projected economic potential. The broader context for developing the process, and the reasons why it failed, have been obscured by time, though subsequent developments in copper hydrometallurgy reveal some of the limitations of this early effort.

The roots of this project can be traced to early research into chalcopyrite leaching at Sherritt. In 1969, for instance, a group of researchers led by Vladimir Mackiw patented a hydrometallurgical method for direct leaching chalcopyrite using sulphuric acid at temperatures between 99 and 121 °C and oxygen partial pressure between 200 and 500 psig (between 1,379 and 3,447 kPa).<sup>228</sup> Given that the later Sherritt-Cominco process operated around 70°C, we might presume that this early effort faced the typical challenge of molten sulphur impeding the leaching process.

Collaborative work on the project began in 1971, when the two companies received funding under the Government of Canada's PAIT (Program for Advancement of Industrial Technology). Initial research focussed on a relatively straight-forward sulphuric acid oxidation leach followed by sulphur removal, recovery of various metals, purification, and electrowinning of copper. However, leaching rates in these early experiments were poor. It was therefore decided that an initial activation phase was required to facilitate the removal of the majority of the iron as jarosite. This led to a complicated arrangement incorporating two different activation processes to cover the range of possible ores.<sup>229</sup>

The project was a major investment that cost both companies and the government of Canada more than CAN\$11 million. In 1975, construction began on the pilot plant at the Sherritt nickel refinery in Fort Saskatchewan, with a capacity of nine tonnes per day of copper concentrate from Sherritt's Ruttan and Fox Lake mines.<sup>230</sup> Its purpose included the testing of construction materials, control procedures and operating parameters for a commercial plant, and an environmental assessment.<sup>231</sup> It also sought to determine whether the initial roasting stage could be carried out in an existing multiple-hearth roaster.

Construction of the facility took 12 months, owing in part to a labour dispute.<sup>232</sup> Commissioning began in January of 1976, and the project culminated in a 30-day demonstration run that ended in mid-November, with the testing of the thermal activation section completed in mid-December.<sup>233</sup> Most of the staff consisted of Fort Saskatchewan employees. Four Cominco engineers were also present. The head researchers reported that "women were employed in several sections of the plant."

This was a significant project involving up to 170 full-time workers during its operation, as well as a number of others working in a part-time or advisory capacity. Throughout the process, samples were constantly collected and analysed; by the end of the piloting period, nearly seven hundred analyses were undertaken and completed each day. Data analysis was carried out using a PDP 11 computer furnished with 128 analogue inputs that were scanned every 10 seconds.<sup>234</sup> This data gathering produced a 19-page, daily report. In early 1978, researchers and other company officials completed a detailed account of the process and its potential, after which nothing further appears to have been published.

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<sup>226</sup> Swinkles and Berezowski 1978, 107, 117.

<sup>227</sup> Swinkles and Berezowski 1978, 105.

<sup>228</sup> Mackiw, Veltman, and Vizolyi, 1969.

<sup>229</sup> Swinkles and Berezowski 1978, 107.

<sup>230</sup> Swinkles and Berezowski 1978, 112.

<sup>231</sup> Kawulka et al. 1978, 122-123.

<sup>232</sup> Swinkles and Berezowski 1978, 105.

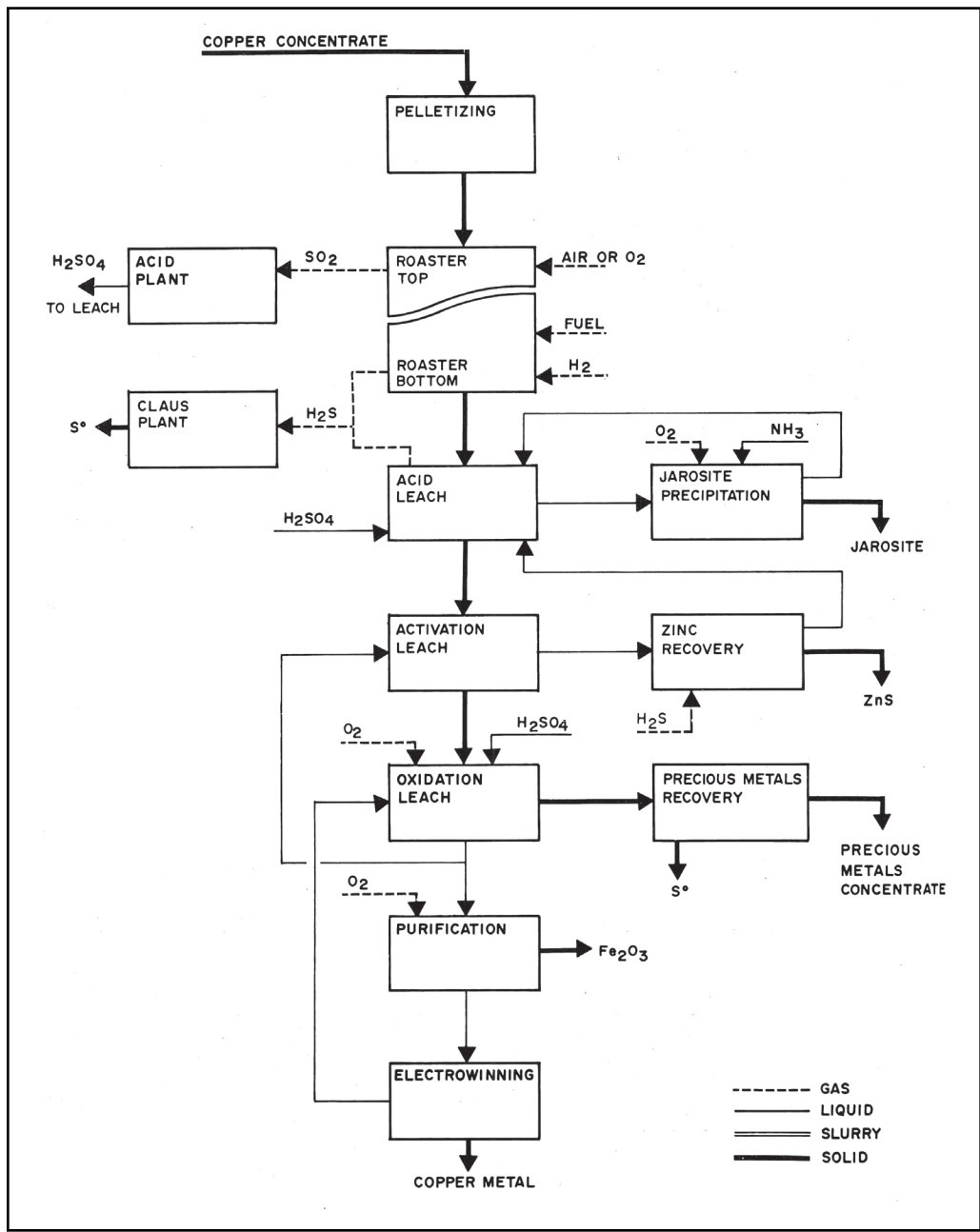
<sup>233</sup> Kawulka et al. 1978, 122, 123.

<sup>234</sup> Kawulka et al. 1978, 123.

# THE SHERRITT-COMINCO FLOWSHEET

Because the process was highly complex, and proceeded no further than an initial piloting stage, numerous aspects and outcomes remain hypothetical. For instance, the initial activation stage to facilitate the removal of iron was imagined as two "front end" possibilities, one an activation leach for low-iron concentrates, the other a roasting step for higher-iron concentrates. Both options could, in theory, be incorporated into a single versatile plant.<sup>235</sup>

Only one of these two options, the activation leach approach meant to be applied to high-copper bornitic concentrates, was entirely hydrometallurgical. Since this did not efficiently dissolve pyrite, a common iron oxide, it was not effective on the leaner, iron-rich chalcopyrite copper ores. Activation leaching began with finely ground ore with leach with copper sulphate recycled from the subsequent oxidation leach step. A replacement reaction produced a copper-rich solid residue to be passed onto the oxidation leach, while the bulk of the iron and zinc passed into a ferrous sulphate solution.<sup>236</sup>



**Figure 12:** Simplified diagram showing the roasting front-end option of the Sherritt-Cominco copper process (Swinkles and Berezowski 1978, 110).

<sup>235</sup> Swinkles and Berezowski 1978, 106, 109.

<sup>236</sup> Swinkles and Berezowski 1978, 107.

The second option involved a preliminary thermal pre-treatment of a pelletized concentrate using a conventional multiple-hearth roaster to drive off over half of the sulphur. The heated pellets would then be treated in a reduction section of the roaster that used hydrogen gas to further remove sulphur. The sulphur dioxide formed in this process would be made into sulphuric acid at an acid plant.

Part of this acid would be used in the subsequent leaching step in which ferrous sulphide would be leached with sulphuric acid at 70°C to remove iron as an iron sulphate solution. In both front-end options, the iron rich solution would be treated at high temperature and pressure in an autoclave, to precipitate iron as jarosite.<sup>237</sup> The solids would be separated and sent to a tailing pond as a jarosite slurry.

The residue produced by the roasting and acid leaching would be transferred to an autoclave for an activation leach similar to the alternate front-end option.<sup>238</sup> There, it would be treated with a copper sulphate solution that would replace much of the remaining iron, and about half of the zinc, with copper-rich residue. The iron and zinc would pass into the leach liquor as sulphates. The leach liquor would be treated with hydrogen sulphide to precipitate the zinc as zinc sulphide, which would be used to produce zinc sulphide powder. This would be sold for further processing.<sup>239</sup>

The residue from the activation leach of either front-end process would then go to an oxidation leach whose primary purpose was to convert sulphide to elemental sulphur and to dissolve the vast majority of the copper to a sulphuric acid solution suitable for purification and metal production.<sup>240</sup> This was developed as a two-stage countercurrent leach involving oxygen injection.

Molten sulphur would be separated from the gangue minerals and removed through flotation and filtration. The remaining sulphur would be removed from the filter cake using solvent extraction.<sup>241</sup> The residue could be further processed to extract molybdenum. A precious metal concentrate could also be produced through several processes including solvent extraction.

Before the leach liquor would enter the final electrowinning stage, it would pass through a purification process in two titanium-lined autoclaves. This would remove trace amounts of materials such as selenium, tellurium, iron, and bismuth that would otherwise lower the efficiency of the electrowinning process.<sup>242</sup> The electrowinning circuit incorporated existing commercial technologies to electrolyze copper onto titanium cathodes from the solution. An alternative approach to metal production, a hydrogen-reduction step to produce copper powder, was also tested at a laboratory scale.<sup>243</sup>

## EVALUATING THE SHERRITT-COMINCO PROCESS

By early 1978, the developers of the Sherritt-Cominco process were able to provide preliminary estimates for a hypothetical 75,000-tonne-per-year facility processing medium-to-low grade chalcopyrite concentrate from Manitoba.<sup>244</sup> The reasons the facility was never constructed have not been published, though its limitations may be glimpsed through this preliminary report. The process was compared primarily with the Outokumpu flash smelter, which was, and remains, the primary pyrometallurgical furnace for smelting copper concentrate.

The main differences between the two approaches lay in their energy requirements. The Sherritt-Copper process was estimated to require nearly twice as much fuel, and three times as much electrical power, as a flash smelter. Beyond the energy benefits of burning sulphur inherent in smelting, the “electrorefining” process used in the conventional pyrometallurgical plant required ten times less voltage than the “electrowinning” process of depositing copper from the purified leach solution. Electrorefining uses cast metal anodes of relatively impure metal to produce cathodes of refined metal through the electrolytic process.

This method, like other hydrometallurgical processes at the time, was notably constrained by the formation of molten sulphur that tended to occlude unleached sulphides as temperature increased. Lower temperatures, however, made for longer leaching times.<sup>245</sup> Its leaching temperature of 70°C was much lower, for instance, than the 150°C of the later Dynatec process, described below, which used a surfactant to inhibit this occlusion process.<sup>246</sup>

There were other minor disadvantages as well. For instance, the process precipitated iron as jarosite, an unsaleable sulphate mineral of iron and potassium that would need to be discarded. Among various possible improvements to the process, the developers speculated that further work could permit the production of hematite, an iron oxide mineral more amenable to further processing.<sup>247</sup>

Metallurgists describing the process have suggested that its limitations likely had much to do with its complexity. Fathi Habashi noted in his review of hydrometallurgical copper processes that: “Evidently the process is complicated

<sup>237</sup> Maschmeyer, Milner, and Parekh 1978, 132; Swinkles and Berezowski 1978, 108.

<sup>238</sup> Swinkles and Berezowski 1978, 117.

<sup>239</sup> Maschmeyer et al. 1978, 132; Maschmeyer, Milner, and Parekh 1978, 127.

<sup>240</sup> Maschmeyer, Milner, and Parekh 1978, 128.

<sup>241</sup> Swinkles and Berezowski 1978, 111.

<sup>242</sup> Maschmeyer, Milner, and Parekh 1978, 128.

<sup>243</sup> Swinkles and Berezowski 1978, 120.

<sup>244</sup> Maschmeyer, Milner, and Parekh 1978, 131.

<sup>245</sup> Swinkles and Berezowski 1978, 118-119.

<sup>246</sup> Stikma et al 1999, 291.

<sup>247</sup> Maschmeyer, Milner, and Parekh 1978, 137.

and there are more obvious and direct routes to get elemental sulphur and copper from chalcopyrite.”<sup>248</sup> Perhaps the greatest outcome of this research was the collaboration itself. While working on this copper process, advances were made at Sherritt in the pressure leaching of zinc that ultimately led to the successful zinc pressure leach process.

Later developments in the hydrometallurgy of copper would address several of the limitations faced by the researchers behind Sherritt-Cominco process. Much of this development took place in the context of the Hudbay facility in Flin Flon, Manitoba, which was still more remote than the Trail plant, and faced many of the same challenges.

## MODERNIZING COPPER OPERATIONS AT HUDBAY

In 1983, the federal Department of Energy, Mines and Resources (now Natural Resources Canada) funded a survey of Canada’s nickel and copper industries. Its purpose was to

improve the industry’s competitiveness while addressing the problem of sulphur dioxide emissions. Released the following year, the report raised concerns about the state of the Hudson Bay Mining and Smelting Company (HBMS, now HudBay) copper-zinc metallurgical complex at Flin Flon, Manitoba. The Flin Flon copper smelter, commissioned in 1930, was then among the oldest in the world. Its pyrometallurgical furnaces, especially the antiquated multi-hearth roasters, were energy-intensive, inefficient, and highly polluting.<sup>249</sup>

The plant produced 70,000 tonnes of copper per year, using the common roast-leach-electrowin method, as well as 80,000 tonnes of zinc per year through zinc fuming, acid leaching, and electrowinning.<sup>250</sup> Neither the 167,000 tonnes per year of toxins emitted by its copper plant, nor the 85,000 tonnes per year from its zinc plant, were captured; the older generation furnaces did not generate the concentrated exhaust streams that permitted the efficient capture of sulphur dioxide. The plant was also far from potential markets for sulphuric acid.<sup>251</sup>



**Figure 13:** A view of the Flin Flon plant in 1972 (FFCA Disk No 12 Open Collection 016, Courtesy of the Flin Flon Heritage Project)

<sup>248</sup> Habashi 2007, 10.

<sup>249</sup> Canada 1983, 78.

<sup>250</sup> Craigen et al. Paper 7, 2.

<sup>251</sup> Canada 1984, 64-65.



Such conditions tended to favour a hydrometallurgical solution, especially on the zinc side, since Sherritt's pressure leaching for zinc was, by then, a proven technology. As noted, the first stand-alone zinc pressure leaching plant in the world would be commissioned at Hudbay's Flin Flon operations in 1993. While this eliminated the sulphur dioxide emissions from the zinc operations, the reverberatory furnaces used in the copper smelter remained a major source of pollution. Numerous options were explored on the copper side, both pyrometallurgical and hydrometallurgical.<sup>252</sup> Two of the hydrometallurgical options that were considered for the plant are described below.

Ultimately, no solution was implemented for Hudbay's polluting copper plant. The company evidently decided on a pyrometallurgical route to modernizing the copper operation, and a Noranda reactor vessel was shipped to the plant site in the early 1990s. This was never installed. Cost overruns in implementing the new zinc plant may have sapped funding needed for the copper modernization. No doubt, other factors also played a role. The outdated copper smelter continued to operate until 2010, when it was finally closed after 80 years of service. The Flin Flon facility continues to operate as a modern zinc plant with copper concentrate processed elsewhere.<sup>253</sup>

## DEVELOPMENT OF THE GREAT CENTRAL MINES (GCM) PROCESS

Following the release of the 1983 government report, several options were explored for modernizing the Hudbay Flin Flon copper operations. Studies by CANMET and Hudbay, conducted over the mid-1980s, considered several technologies, including the Inco flash furnace, the Noranda reactor, and the chloride-based hydrometallurgical GCM process. Flash smelting was identified as the preferred short-term option, while the GCM process was the preferred long-term alternative. The latter matched a number of the conditions discussed in the report for a hydrometallurgical solution, including the elimination of sulphur dioxide emissions and a capacity to generate elemental sulphur. By the time that this decision was being made, the Sherritt zinc pressure leach process had been chosen for implementation.<sup>254</sup>

The technology considered for Hudbay was a modification of a recently developed hydrometallurgical process meant for higher-grade ores than those processed at Flin Flon. Development had begun in 1976, when Great Central Mines of Vancouver commissioned Bacon, Donaldson, and Associates, a Vancouver-based engineering company, to investigate a novel electrowinning process for copper. This was developed into a complete process for processing high-grade porphyry copper concentrates.<sup>255</sup>

A U.S. patent was granted for this process in 1979.<sup>256</sup> After further laboratory testing, Great Central Mines requested that the process be evaluated by CANMET. Following a positive assessment, further development was carried out by Bacon Donaldson over the early 1980s. This led to an improved process, and a renewed set of patent applications submitted in 1984.

Based on these promising beginnings, it was decided to pursue testing and development of a modified process suitable to the conditions of the Hudbay plant. The Hudbay copper concentrates were lower in copper, and higher in zinc and lead, than those for which the process had been developed.<sup>257</sup> They also contained levels of precious metals that were efficiently recovered under the existing process. Its replacement would have to effectively recover gold and sulphur from the tailings.<sup>258</sup>

<sup>252</sup> Stikma et al. 1999, 290.

<sup>253</sup> Mackey and Davenport 2011, 116-117.

<sup>254</sup> Craigen et al 12.

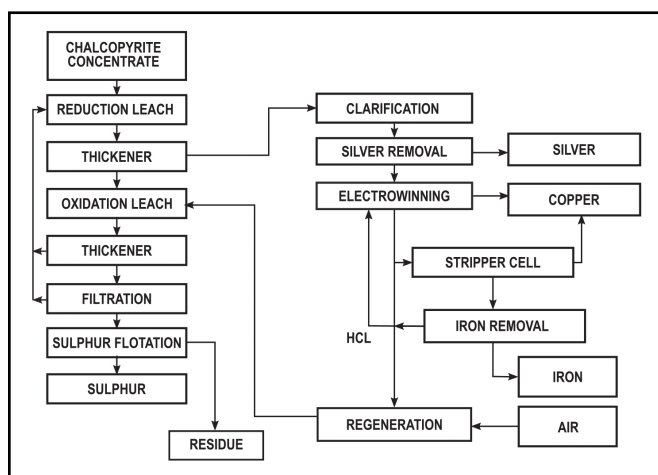
<sup>255</sup> Beattie et al. ; Craigen et al. Paper 7, 4.

<sup>256</sup> Bacon and Vreugde 1979.

<sup>257</sup> Beattie, Craigen, and Sarkar 1984, 22.

<sup>258</sup> Dutrizac 1992 11.

The GCM process, as developed for HudBay, began with an initial grinding of copper concentrate to a fine powder. A preleach step using hydrogen chloride removed pyrrhotite (an iron sulphide mineral), zinc and lead. This leach solution was sent to an evaporation and oxydrolisis step to remove iron as hematite, and remaining metals as sulphates. Hydrochloric acid was recovered from this operation and recycled into the process.<sup>259</sup>



**Figure 14:** Flowsheet of the GCM Process [Image based on Craigen et al. p. 3]

The solids from the preleach step entered the leaching phase, where they were treated at atmospheric pressure with ferric chloride and salt. This process dissolved copper and silver into solution while converting sulphides to elemental sulphur. This was followed by a second oxidizing leaching stage to optimize metal extractions. Silver was removed before electrowinning took place. Copper in this process was produced in diaphragm cells as a granular material.<sup>260</sup> The leach solution underwent a regeneration process during which iron was removed. Elemental sulphur was recovered from the final leach residue by hot pressure filtration at 150°C.<sup>261</sup>

Estimates based on these tests found the development costs of the GCM process to be higher than those of the Noranda or Inco flash smelter.<sup>262</sup> At the time that it was considered, a method for recovering gold and silver from the sulphide filter cake residue had not yet been developed. This would have required a much more efficient method for filtering sulphur in order to produce a concentrated sulphide remainder that could either be processed onsite at a purpose-built facility or sold. Offsite processing would have cut existing revenue.<sup>263</sup>

The process would have required considerable piloting and optimisation in order to produce copper that was as pure as the existing method.<sup>264</sup> The recovery of elemental sulphur was lower than other ferric chloride base processes; it recovered around 60 to 70 percent of sulphur while leaving a large sulphate remainder. Generally speaking, the GCM process likely represented a significant investment towards an uncertain end. It was still under development into the early 1990s.<sup>265</sup>

## DEVELOPMENT OF THE DYNATEC COPPER PROCESS

The Dynatec process was developed in large part with the goal of replacing smelting facilities at Flin Flon. It is considered an “additive enhanced pressure leaching” process because it employs a surfactant additive that permits the chalcopyrite to react with the leach in the presence of molten elemental sulphur.<sup>266</sup> The process is similar to the earlier zinc process in several ways, including its production of elemental sulphur rather than sulphuric acid.

The partnership between Dynatec and Hudbay emerged following the implementation of the novel two-stage pressure leaching for zinc discussed in the previous chapter. Dynatec, a mining company founded in 1980, acquired Sherritt International Consultants in 1997, when Sherritt divested a part of its technical division during its transition to Cuban concentrates. Sherritt reacquired Dynatec in 2007 for CAN\$1.6 billion.<sup>267</sup>

By 1997, test work had proceeded to the point where Dynatec could present various possible flowsheets to representatives of Hudbay.<sup>268</sup> As with the GCM process, the effective recovery of precious metals at the existing Flin Flon facility required that the new process recover gold and silver from the oxide tailings at a comparable or better rate.

Dynatec proceeded with laboratory test work through the fourth quarter of 1997. The leach solution proved amenable to solvent extraction by a common extractant. Tailings were verified for compatibility with standard carbon-in-pulp (CIP) cyanide leaching of the pressure leach residue.<sup>269</sup> Cyanide leaching yielded 95 percent gold and 99 percent silver extraction. On this basis, it was decided to proceed to the development of a conceptual flowsheet for HudBay.

<sup>259</sup> Craigen et al. Paper 7, 5.

<sup>260</sup> Dutrizac 1992, 11.

<sup>261</sup> Craigen et al. Paper 7,7.

<sup>262</sup> Dutrizac 1992, 11.

<sup>263</sup> Craigen et al 15-18.

<sup>264</sup> Craigen et al. Paper 7, 5, 9.

<sup>265</sup> Dutrizac 1992, 11.

<sup>266</sup> Collins, Michael. (December 7, 2018) Personal Phone Interview. 22:35.

<sup>267</sup> Grant 2007.

<sup>268</sup> Stikma et al 1999, 291.

<sup>269</sup> Stikma et al 1999, 291.

# THE DYNATEC/HBMS PROCESS FLOWSHEET

In the Dynatec process, copper concentrate is leached in an autoclave at elevated temperature and pressure alongside recycled sulphides from the later sulphur filtration and separation step. Leaching is conducted in a sulphuric acid medium. Copper extraction is limited to between 80 and 90 percent at this stage, in order to limit the leaching time. Because the leach is conducted at a moderate temperature, sulphide is converted primarily to elemental sulphur.<sup>270</sup>

The leach residue undergoes flotation to separate a concentrate of elemental sulphur and unleached sulphide from an oxidic tailing. The sulphur-sulphide concentrate is melted and filtered, and the elemental sulphur is stockpiled or sold. The sulphide is returned to the leach to recover the remainder of the leachable copper, and to return a large portion of the precious metals back into the system where they will report to the tailings.

The tailings are processed in a precious metals recovery system based on the conventional cyanide pressure leach and carbon-in-pulp recovery.<sup>271</sup> The tailings undergo a lime boil “silver enhancement treatment” prior to cyanide processing, in order to improve silver recovery.<sup>272</sup> Precious metals are

recovered using either cementation with zinc powder or electrowinning. The product of this process, a cast bar of doré metal (an unrefined mixture of gold and silver exceeding 95 percent purity) would have been sold for further refining.

The pregnant solution, consisting of about 40 grams per litre of copper, 5 grams per litre of iron and 20 grams per litre of sulphuric acid is treated using solvent extraction. The pregnant solvent is electrowon to produce cathode copper. Electrolyte from the electrowinning step is returned to the solvent extraction stage to strip the pregnant solvent. Most of the raffinate is recycled to the initial pressure leaching step. A balance of zinc and sulphates is maintained through a bleed stream of raffinate that is neutralized using limestone or lime, and processed to remove iron, copper and zinc. Precipitated iron and copper from this process are returned to the pressure leaching circuit, while the remaining zinc solution is treated with lime to precipitate zinc sulphate that could be processed in the zinc plant.<sup>273</sup>

The Dynatec process has gone through several pilot plant campaigns, but was not adopted at Hudbay. No commercial application has yet been found. Its principal disadvantage is likely shared with other hydrometallurgical copper processes: that is, the need to find a use for the sulphuric acid remaining from high-temperature extraction, for example, to leach low-grade ore.<sup>274</sup>

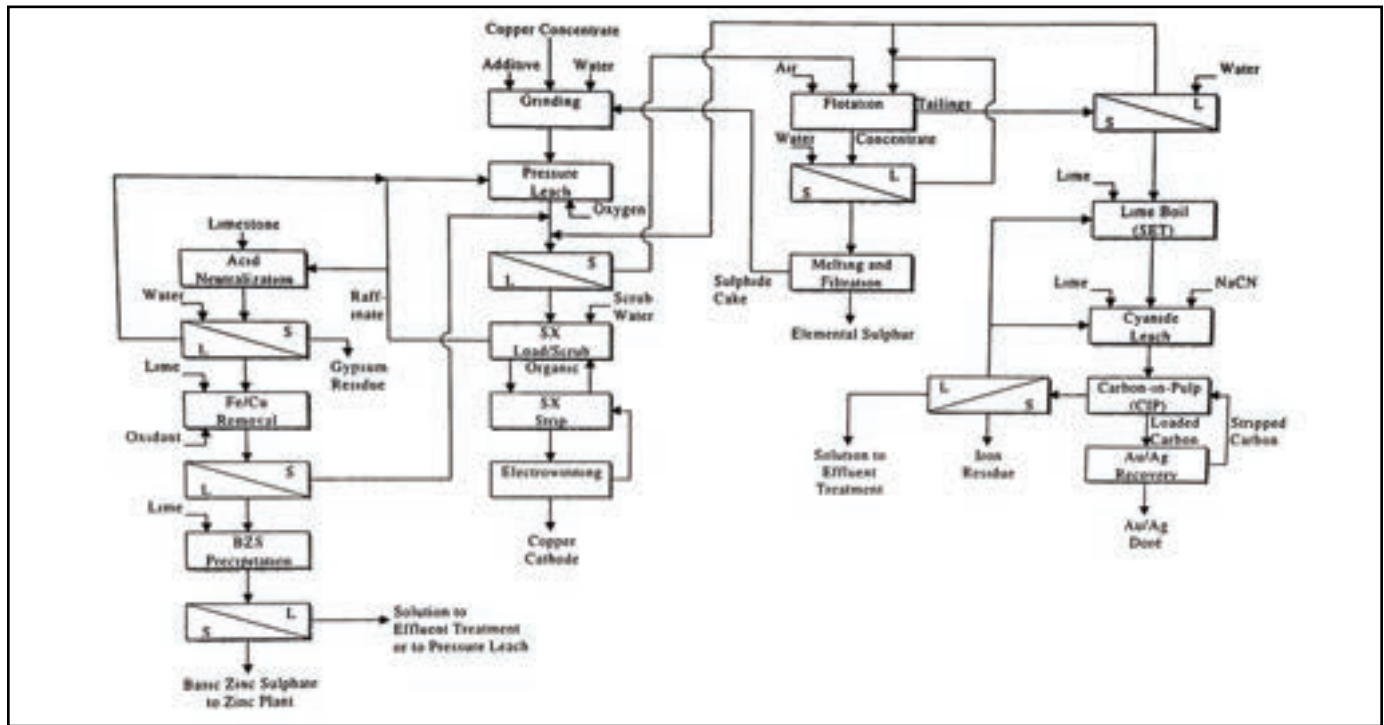


Figure 15: Dynatec’s copper pressure leach process flowsheet proposed for HBMS/Hudbay (Stiksma et al 1999, 293).

<sup>270</sup> Stiksma et al 1999, 292.

<sup>271</sup> Stiksma et al 1999, 292.

<sup>272</sup> Stiksma et al 1999, 299.

<sup>273</sup> Stiksma et al 1999, 294.

<sup>274</sup> Collins, Michael. (December 7, 2018) Personal Phone Interview. 22:40.

## DEVELOPMENT OF THE CESL PROCESS

The Cominco Engineering Services Limited (CESL) process is a “chloride assisted” acid leach. It is not clear whether it was ever seriously considered as an option for the Hudbay copper operation. However, it is arguably the most extensively developed of the copper processes described here.

Cominco Engineering Services Limited (CESL) began as the engineering department of Cominco of Vancouver, British Columbia. In the early 1980s, it was developed into an engineering, procurement and construction management (EPCM) company owned by Cominco. The company was responsible for a number of engineering projects, including the Cominco zinc plant, and a new electrowinning plant at Trail that was installed in 1981. It also developed novel technologies, including an electrolytic lead process that it sold to Hindustan Zinc in 1987.<sup>275</sup>

Development of the CESL process began in 1992 as the “Refined Metal Project” that sought a hydrometallurgical alternative for copper sulphide. It was designed to incorporate existing engineering technologies in an innovative manner to create a flexible process applicable to a range of concentrates. It would accommodate low-grade concentrates with high impurities, and produce environmentally stable residues, all with no sulphur dioxide emissions. It was also designed to be cheaper to implement than conventional smelting. The CESL process incorporates a solvent extraction step to efficiently extract a purified and concentrated solution from the pregnant leach.<sup>276</sup>

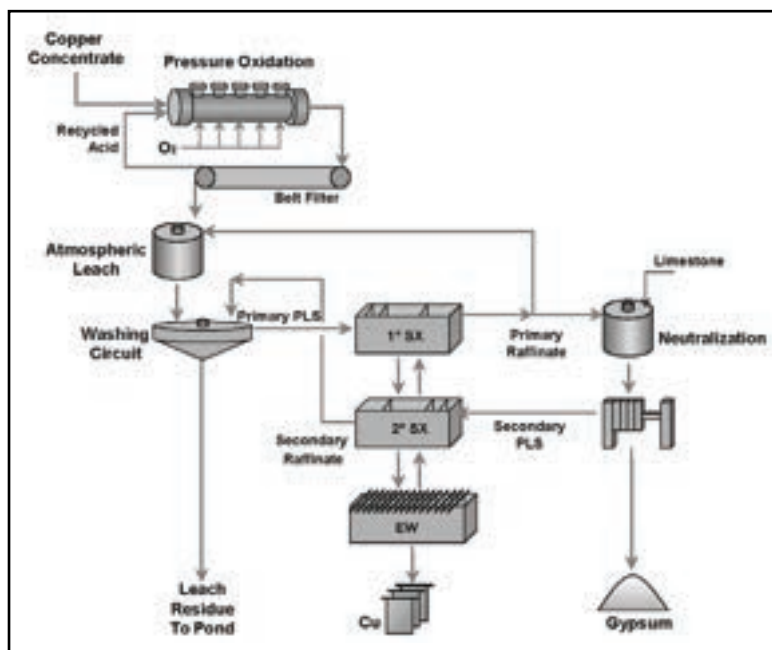
Successful bench-scale tests led, in 1994, to the opening of a test plant with a capacity of 13 tonnes per year of cathode copper. In 1996, a much larger demonstration-scale plant with a capacity of around 500 tonnes per year of cathode copper was designed in order to test the engineering concepts and to reduce the risks of scaling up the process. This operated from 1997 to 2000, and was tested on a variety of copper concentrates.<sup>277</sup>

This process was developed for implementation at the Highland Valley Copper (HVC) mine located in southern British Columbia. Cominco acquired an interest in this massive open pit mine in the mid-1980s, intending to bring the CESL process online in 1997 and 1998. However, this coincided with a downturn in metal prices that made it much cheaper to process HVC concentrate in various existing smelters that were operating below capacity.<sup>278</sup>

Despite this setback, CESL continued to develop the process, which has since evolved to incorporate a number of metals beyond copper. Further testing in the early 2000s was carried out on the processing of copper-zinc and copper-nickel-cobalt concentrates, as well as on the recovery of gold from plant residue using cyanidation. The following is a description of the basic process as it might have been applied in the 1990s.

## THE CESL PROCESS FOR COPPER

The CESL process begins with a fine grinding of the copper concentrate. This produces a liquid slurry that is thickened to 68 percent solids before being pumped to the autoclave for pressure oxidation that is carried out in an autoclave at 150°C and 200 psi (1,379 kPa). The leaching agent is sulphuric acid produced in the electrowinning step mixed with 10 to 12 grams per litre of chloride, which acts as a catalyst.



**Figure 16:** The CESL process for copper (Defreyne, de Souza Costa et al 2004, 4. Image courtesy of David Jones).

<sup>275</sup> Anon. "Businessline" *Journal of Metals* 39, 4-7 July 1987, 4. In 1994, the engineering side CESL was merged with the Vancouver-based engineering company H. A. Simons, while the company's process development assets remained under Cominco. CESL currently operates as part of Teck's technology division. The entity separated in 1994, and eventually became Canadian Process Technologies, which specialized in flotation and flotation equipment. In 2007, Canadian Process Technologies was acquired by Eriez of Pennsylvania and is now the Eriez Flotation Division.

<sup>276</sup> Defreyne et al. 2004, 2.

<sup>277</sup> Defreyne et al. 2004, 2.

<sup>278</sup> Defreyne et al. 2006, 2.

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During pressure oxidation, elemental sulphur is formed, iron is oxidized to hematite, while copper is oxidized to copper sulphate salt that is recovered from the filter cake. A chemical surfactant is used to prevent molten sulphur from occluding the concentrate slurry.<sup>279</sup> Solids are then separated and continue to an atmospheric leach step. Liquids from the separation stage are recycled to the autoclave.<sup>280</sup>

The subsequent atmospheric leaching step dissolves the bulk of the copper into solution. The efficiency of the CESL process comes, in part, from the very limited dissolution of iron into solution during pressure oxidation and atmospheric leaching. This ensures that much of the acid leaching solution can be reused, since a neutralization step to precipitate iron is unnecessary.<sup>281</sup>

Slurry from the pressure oxidation process and the atmospheric leach is washed using neutralized raffinate in a counter-current decantation circuit to recover further copper from the leach residue and to produce a clear solution. The solution is then treated by a two-stage solvent extraction that separates the pregnant electrolyte from a raffinate remainder. About one third of the raffinate from this process is neutralized, and this neutralized solution is further processed through solvent extraction to remove additional copper. The remainder of the raffinate is returned to the leach. The pregnant electrolyte from the solvent extraction process is electrowon in the conventional manner.<sup>282</sup>

## IMPLEMENTATION AND ELABORATION OF THE CESL PROCESS

In 1998, the Brazilian Companhia Vale do Rio Doce (CVRD, now Vale) chose to test CESL process on concentrates from its then-undeveloped Alemão and Salobo properties in the Carajás Mountains of Northern Brazil. Initial trials at CESL facilities in Vancouver, followed by a feasibility study by the Canadian engineering company, Hatch, proved the technical viability of the CESL/CVRD process, as this implementation of the CESL process was known.

In 2005, it was decided to proceed with a small-scale pilot plant with a capacity of 10,000 tonnes per year. Hatch and the Brazilian engineering company SEI Consultoria E Projetos S/C Ltd. conducted the engineering work. Construction began in 2006. The process of contracting local workers, along with the unconventional materials needed to safely contain the chloride-assisted leaching system, led to construction delays. The plant was commissioned in 2008 and was set to run for at least two years.<sup>283</sup> This near-commercial plant provided insight into the materials and design necessary to withstand the highly corrosive chloride leach.<sup>284</sup>

The CESL process has also been adapted to nickel, molybdenum, and gold concentrates, though none of these efforts has yet proceeded past the testing and demonstration stage. In 2010, Teck completed collaborative test work in partnership with the copper producer Aurubis, which is based in Hamburg, Germany. This involved a modification of the copper process meant to capture arsenic as stable ferric arsenate and scorodite within leach residues. As the percentage of copper sources with significant quantities of arsenic increases, and regulation surrounding the treatment of arsenic ores may become stricter, this process may see commercial implementation. Development work is underway to demonstrate the nature and long-term stability of arsenic compounds in the leach residue.<sup>285</sup>

Aurubis and Teck have also collaborated on testing the CESL gold process at a larger scale in order to improve and assess its commercial viability. Given the latter company's focus on copper production, this testing was based on residue produced from a copper concentrate. The process uses pressure cyanidation and a carbon-in-column circuit to capture precious metals from copper plant residue.<sup>286</sup>

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<sup>279</sup> Defreyne and Cabral 2009, 6.

<sup>280</sup> Collins et al 2011, 298; Defreyne et al 2006, 2.

<sup>281</sup> Defreyne et al 2006, 2.

<sup>282</sup> Collins et al 2011, 298; Defreyne et al 2006, 2; Defreyne and Cabral 2009, 4.

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<sup>283</sup> Defreyne et al 2008, 1-2; Defreyne et al 2004, 1.

<sup>284</sup> Defreyne and Cabral 2009, 8.

<sup>285</sup> Bruce et al, 2014, 2-3.

<sup>286</sup> Robinson, Lee, and Heidel 2012, 2-3.

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# PRESSURE OXIDATION OF REFRACTORY GOLD

The cyanidation process for extracting gold is the oldest, and most widespread, process in hydrometallurgy. It is a leaching process that dissolves gold from its ore using an aqueous solution of cyanide. The gold may then be recovered from solution through a number of processes. The most long-lived of these is the Merrill-Crowe method of cementation using zinc powder, though a variety of newer methods have been devised. The carbon-in-pulp (CIP) and the newer carbon-in-leach (CIL) processes are very common today.<sup>287</sup>

Effective cyanidation requires the aqueous solution to contact gold under favourable chemical conditions. As with other metals, the mining industry has increasingly turned to lower-grade ores in which the material is less concentrated or otherwise more difficult to process using traditional methods. Many low-grade ores contain gold particles that are finely disseminated or occluded by other materials—iron and iron-arsenic sulphide minerals are especially common. Such ores are referred to as “refractory.”

The traditional way of processing refractory ores involves pre-treating a sulphide concentrate, produced through flotation, in a refractory furnace—hence the “refractory” name.<sup>288</sup> This converts the sulphur to sulphur dioxide gas while producing a hematite (oxide-rich iron) concentrate that is more amenable to cyanidation. Roasting gold ore carries the typical disadvantages of sulphur dioxide production requiring conversion to sulphuric acid. The roasting of refractory ores containing arsenic also produces toxic arsenous oxide, which is an environmental hazard.<sup>289</sup>

In order to improve the recovery of gold and decrease the environmental footprint of gold operations, a number of new methods have been devised for processing refractory ore. This chapter focusses on two Canadian examples. The first of these derives from the research tradition, emerging from Sherritt, of pressure leaching and pressure oxidation. Pressure oxidation replaces the roasting method of removing sulphides in order to liberate entrapped gold particles.<sup>290</sup>

The pressure oxidation of refractory gold ore takes place in an autoclave at temperatures between 170 and 225°C, and at pressures between 1,100 and 3,200 kPa with oxygen injection. The reaction is exothermic, requiring little

additional heat. This oxidizes the sulphides to sulphate. The degree to which the sulphide mineral is oxidized varies according to the requirements of preparing a particular ore for effective cyanidation. The gold is deposited with the oxidized solids, which are suitable for cyanide leaching.<sup>291</sup> The vast majority of processes use sulphuric acid, though the Canadian mining company Barrick Gold Corporation notably developed an innovative alkaline leach process for preparing carboniferous feeds that would otherwise consume significant amounts of acid.

Depending on the ore, pressure hydrometallurgy may produce other benefits. For instance, the pH of the leaching solution can be adjusted to optimize the cyanidation process. The process may also be used to fix arsenic into a ferric arsenate for safe disposal. Having been adopted at dozens of locations around the world, pressure oxidation for gold is clearly the most successful technology to emerge from the legacy of pressure hydrometallurgy originating at Sherritt.

A second domain of Canadian development in this area has been the successful industrialization of the thiosulfate leaching process by Barrick—the first, and so far only, industrial implementation of this long sought-after process. Thiosulfate leach is only used where cyanide leaching is not viable. This would either be due to environmental reasons—some jurisdictions forbid the use of cyanide—or to treat particular “preg-robbing” gold ores.

Preg-robbing ores contain carbonaceous materials that, if dissolved into the leach solution, will prematurely absorb or “rob” gold (aurocyanide) compounds during the cyanide leaching process.<sup>292</sup> It was the search for a solution for preg-robbing ores that led to the discovery of the CIP process for concentrating gold within the prepared leach solution.<sup>293</sup> Occasionally, ores are both refractory sulphide and contain carbonaceous material—hence “double-refractory.” This is the case with some of the ores from Barrick’s enormous Goldstrike mine in northeastern Nevada, for which their sodium thiosulfate leaching process was developed.

Barrick’s development of thiosulfate leaching resulted from a decision to invest heavily in forward-looking research at a time when other major Canadian mining companies were divesting longstanding R&D facilities. The implementation of both pressure oxidization and pressure leaching required several decades of research and testing before being commercially implemented. Both required the ownership of large bodies of challenging gold ore—respectively, Homestake in northern California, and Goldstrike in Nevada—to justify the associated risks and costs.

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<sup>287</sup> Deschênes et al. 2011, 203.

<sup>288</sup> Berezowsky et al. 1991, 12.

<sup>289</sup> Deschênes et al. 2011, 203.

<sup>290</sup> Thomas 2005, 359.

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<sup>291</sup> Berezowsky et al. 1991, 12.

<sup>292</sup> Ji et al 2003.

<sup>293</sup> Afenya 1991, 1407.

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## Sherritt and the development of acid pressure oxidation (POX) of refractory gold ores

Given the longevity of the hydrometallurgical cyanidation process, it is not surprising that pressure oxidation for treatment of refractory gold ore was explored very soon after the successful implementation of ammoniacal pressure leaching at Fort Saskatchewan. In the mid-1950s, Chemico, which had played a major role in engineering the Fort Saskatchewan plant, developed a series of patents related to the recovery of gold from the residues of pressure-leached cobalt arsenides. Follow-up investigations were then carried out on a number of refractory gold ores.<sup>294</sup>

Sherritt also examined the processing of ores containing iron-arsenic sulphides in the 1950s. The investigations at Sherritt and Chemico both showed improved yields from cyanidation following initial pressure oxidation. When, in 1956, Sherritt purchased Chemico's patents in pressure leaching, it consolidated a great deal of existing research relating to gold.<sup>295</sup>

However, this process would not find an application until the early 1980s, when the industry's increasing turn towards refractory ores led Sherritt to restart work on the process. The Sherritt pressure oxidation (POX) process for refractory gold ore was developed in collaboration with the Homestake Mining Company in order to process ore from

the McLaughlin Mine in the Lake Country of northern California. The complex ore body had been discovered in 1979, and was to become the largest gold deposit discovered in California in the twentieth century. During its operation between 1985 and 2002, the mine would produce about a billion dollars' worth of gold.<sup>296</sup>

In search of a means to process this challenging ore, Homestake enlisted two American companies, Dawson Metallurgical of Salt Lake City, Utah, and Hazen Research of Golden, Colorado, to perform metallurgical testing. It was found that the ores responded poorly to conventional cyanidation. In 1980, tests at Dawson Metallurgical determined that acid pressure oxidation of flotation concentrate could lead to the extraction of 90 percent of gold values through cyanidation. In June of 1981, Homestake approached Sherritt-Gordon to confirm previous results and to develop pilot-scale testing facilities. Tests at Sherritt on the McLaughlin concentrate confirmed the viability of pressure oxidation.<sup>297</sup>

An extensive piloting program at Fort Saskatchewan gathered data from a four-compartment laboratory autoclave. Construction on the McLaughlin processing plants began in 1983. The pressure oxidation autoclave began operation in July of 1985.<sup>298</sup> A conference report given two years later by technicians from Homestake reported that the process was operating well. The following describes the leaching process at McLaughlin as it was implemented at that time.

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<sup>294</sup> Berezowsky and Weir 1984, 1.

<sup>295</sup> Berezowsky and Weir 1984, 1.

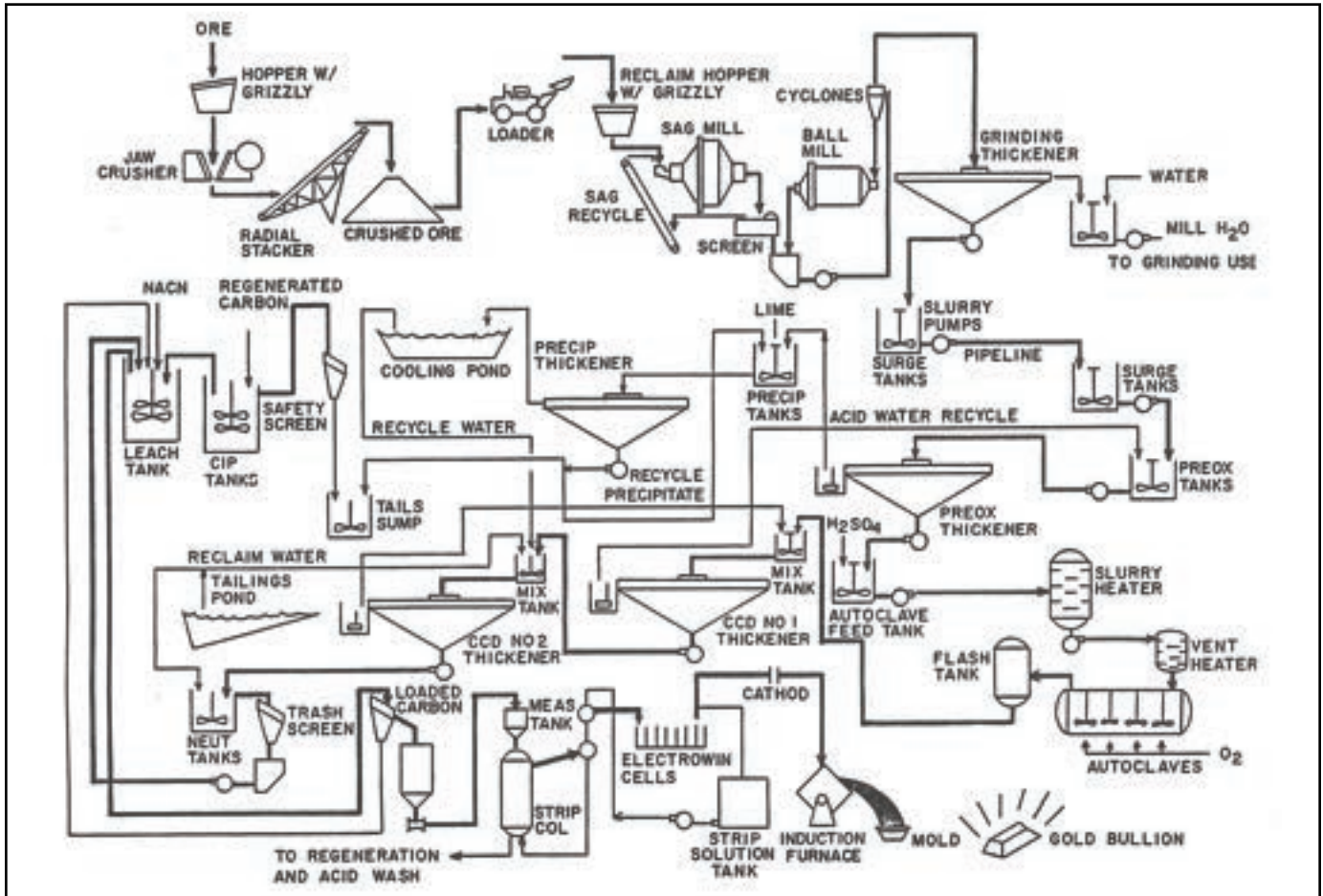
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<sup>296</sup> Nolte 2003.

<sup>297</sup> Turney, Smith, and Janhunen 1989, 31.

<sup>298</sup> Turney, Smith, and Janhunen 1989, 25-31.

# THE PRESSURE OXIDATION AT MCLAUGHLIN MINE



**Figure 17:** Pressure oxidation flowsheet for the McLaughlin mine (Turney, Smith, and Janhunen 1989, 38).

The process of extracting gold from McLaughlin refractory ore began at the mine site where ore was crushed then ground to a fine and consistent powder using a series of milling machines. This produced a slurry of 40 to 45 percent solids that was pumped 7.24 kilometres (4.5 miles) to the processing facility.

This slurry was treated in a preoxidation step using an acid leach of recycled sulphuric acid in order to remove impurities. The slurry was thickened and transferred to an autoclave where the ore was processed for about 90 minutes. Leaching was carried out at 160°C under oxygen injection until roughly 85 percent of the sulphides had been leached. The level of oxidation was determined by measuring the

electrical potential of the leaching liquid.<sup>299</sup> Once leaching was complete, the slurry was washed, cooled, and thickened.

Acid from the thickening process was recycled to the preoxidation step. Thickened solids were neutralized using milk of lime and diluted with water in preparation for the cyanidation process. This took place in two parallel systems, each of which involved one cyanide leaching step followed by five stages in which the leach solution was exposed to carbon in order to absorb the gold from the leach solution. The loaded carbon was stripped using a caustic solution and was regenerated for reuse. Gold and silver were electrowon and the cathodes were smelted to produce a doré product (about 80 percent gold mixed with silver) that was sold for further purification.<sup>300</sup>

<sup>299</sup> Turney, Smith, and Janhunen 1989, 39.

<sup>300</sup> Turney, Smith, and Janhunen 1989, 37.



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## GOLD PRESSURE OXIDATION FLOURISHES

The McLaughlin mine passed to Barrick when Homestake was purchased in 2001. It continued to operate until 2002. It has since been reclaimed as the Donald and Sylvia McLaughlin Natural Reserve. The success of pressure leach process pioneered at McLaughlin is evident in its subsequent widespread international adoption.

In 1986, the year after operations commenced at McLaughlin, the much smaller São Bento pressure oxidization facility was opened in Brazil. Developed by Sherritt, this facility operated until 2007. AGA Brazil purchased the facility in 2008 from Eldorado in order to transform it into a modern hydrometallurgical plant for concentrate from the Córrego do Sítio gold complex. Sherritt technologies carried out the development on this pressure oxidation project, which opened in 2012.<sup>301</sup>

Between 1985 and 1991, commercial pressure leach plants were opened in Canada, the United States, Brazil, Greece, New Guinea.<sup>302</sup> By 2012, there were more than thirty,<sup>303</sup> Canadian engineering and metallurgical companies, including Hatch, SNC-Lavalin, and SGS Lakefield, were involved with most of these projects.<sup>304</sup>

One important Canadian development in this field has been the recent industrial application of alkaline pressure leach by Barrick. First applied at the Merkur mine in Utah in 1988, this was later implemented at Barrick's enormous Goldstrike property. Alkaline leach makes it possible to treat carbonaceous refractory ores that would neutralize the standard sulphuric acid medium resulting in uneconomical levels of reagent use. While carrying various disadvantages that make it unsuitable for most ores, it is less corrosive than acid leach and eliminates the neutralization step.<sup>305</sup> The transition to alkaline leach at Goldstrike prolonged the career of the mine's autoclaves. This allowed for the eventual adoption of thiosulfate leaching, which is discussed below.<sup>306</sup>

## BARRICK GOLD'S CALCIUM THIOSULFATE LEACHING PROCESS AT GOLDSTRIKE MINE

In 2015, Barrick Gold opened the first commercial-scale thiosulfate leaching operation at its flagship Goldstrike mine in northeastern Nevada. This was a significant milestone in a field dominated by the longstanding cyanide leaching process. Whereas pressure oxidation is used to prepare refractory gold ores for the conventional cyanide leach/CIP method, the thiosulfate approach replaces that method entirely. When compared to cyanide, thiosulfate appears to have little to recommend it. Notably, thiosulfate tends to oxidize so rapidly that the process consumes an uneconomical quantity of lixiviant.<sup>307</sup>

However, thiosulfate has two fundamental advantages: it is relatively benign compared to cyanide, a notoriously poisonous chemical. Cyanide has been involved in several significant mining accidents, some involving Canadian mining companies operating abroad.<sup>308</sup> This is a somewhat theoretical advantage since relatively few gold-producing jurisdictions have banned cyanide, which is a highly regulated and extremely efficient lixiviant for gold.<sup>309</sup>

Nevertheless, the calcium thiosulfate lixiviant used in the Barrick process is a comparatively benign chemical that is commonly used as a potato fertilizer. Whereas the vast majority of earlier research on thiosulfate had focussed on ammonium thiosulfate, calcium thiosulfate has environmental advantages, incurs less wear on the process equipment, and is better for the operators because it does not produce the noxious odour of ammonia.<sup>310</sup>

Thiosulfate's primary advantage is its application to double-refractory gold ores—ores that are both sulfidic and carbonaceous. Highly carbonaceous ores cannot be processed directly using the conventional cyanide method because the carbon in the ore absorbs (or "robs") gold from the leach solution. This can be addressed by roasting the ore. Alternately, if a hydrometallurgical oxidization step is used, carbonaceous material can be destroyed using a pre-treatment step incorporating recycled acid. If this is not viable, carbonaceous ores can be treated by switching to an alkaline oxidization step that is not inhibited by carbon.<sup>311</sup>

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<sup>301</sup> Collins et al. 2012, 3.

<sup>302</sup> Berezowsky et al 1991, 14.

<sup>303</sup> Collins et al. 2012, 4.

<sup>304</sup> Collins et al. 2011, 304.

<sup>305</sup> Dani and Papangelakis 2012, 54.

<sup>306</sup> Afenya 1991, 1051.

<sup>307</sup> Muir and Aylmore 2004, 547-550.

<sup>308</sup> Jamasmie, 2017.

<sup>309</sup> Choi et al. 2009.

<sup>310</sup> Kondos, Peter. (December 18, 2018) Personal Phone Interview. 21:00.

<sup>311</sup> Thomas 2005, 357.

However, certain highly carbonaceous or copper-gold ores cannot be effectively treated using oxidization followed by cyanide leach, either because of low recovery rates or unacceptably high levels of reagent use.<sup>312</sup> Thiosulfate's low affinity for carbon has made it a key area of research in pursuit of an alternative lixiviant to cyanide. This process precludes the use of the standard carbon-based process for recovering gold into a concentrated solution. As an alternative, Barrick's process uses ion-exchange resin throughout the leaching process.<sup>313</sup>

The development of thiosulfate leach at Barrick resulted from a confluence of several factors. The company's Goldstrike mine is a large enough gold deposit to justify the effort and expense to bring these marginal ores into production. The mine's roasters were operating at full capacity, while its autoclaves, which had previously been adapted to the alkaline pressure oxidization process, had run through the stock of useable ore. If a new use could be found, that infrastructure, representing a considerable sunk cost, would remain productive. Finally, the company had reacquired patents for thiosulfate leaching through a merger. A prescient investment in research and development would provide the impetus needed to successfully apply this process.

## DEVELOPMENT OF BARRICK'S THIOSULFATE LEACHING PROCESS

Thiosulfate has been a well-studied process among the various alternative lixivants for gold. This research includes a great deal of Canadian work. Several patents cite the research carried out in the late 1970s and 1980s at Sherritt. This work was focused on Manitoba chalcopyrite processed at Fort Saskatchewan. These copper ores contained significant amounts of gold and silver, which could be economically recovered from the leach residue.<sup>314</sup> This process used an ammonium thiosulfate lixiviant and precipitated gold from solution using zinc dust.<sup>315</sup>

Barrick's development of this process followed a somewhat circuitous path. It resulted from the company's acquisition, in 1986, of the Goldstrike mine in Nevada, still among the largest goldmines in the world. In 1990, the first pressure oxidization plant came online at the mine to process refractory ores. The following year, the company began to stockpile low-grade carbonaceous double-refractory ore.

During this period of expansion in the 1990s, Barrick began to experiment with thiosulfate leaching as an option

for processing its growing stockpile of carbonaceous ores. Patents from the mid-to-late 1990s, for a process involving pressure oxidation followed by thiosulfate leaching and resin-in-leach recovery, likely represent this early stage of research.<sup>316</sup> This research introduced a fundamental aspect of the process: the use of a resin system to quickly absorb thiosulfate during the leaching process in order to prevent it oxidizing.<sup>317</sup> As it happened, Barrick chose to expand its roasting facilities instead. The roaster was completed in 2000, at a cost of US\$330 million.<sup>318</sup>

The same year, Barrick sold its existing patents in thiosulfate leach to Placer Dome, which continued to develop the process at its research facility in Vancouver. At this point, Placer Dome was in competition with the Newmont Mining Corporation, based in Colorado, to develop a thiosulfate-based process. Both rigorously defended their intellectual property claims. Newmont would eventually take its own process to the demonstration stage before halting development. In 2005, Barrick acquired Placer Dome. In doing so, it reacquired the IP rights to the thiosulfate process along with the Placer Dome intellectual property lawyers, and the Vancouver-based Placer research group.<sup>319</sup>

The reacquisition of thiosulfate technology occurred at a point at which the company had decided to commit itself to research and development. Around 2003, the company had launched the Barrick Strategic Technology Solutions group. In a recent interview for this report, Peter Kondos, formerly senior director of Strategic Technology Solutions at Barrick, attributes this initiative to Greg Wilkins (1956–2009) then-president and CEO of Barrick. Kondos notes:

Greg Wilkins was a visionary. He wanted to develop the technology group—the R&D group as he called it at that time—as a dynamic group that creates value for the company like exploration does. He said that, “We don't know what exploration will come up with. Sometimes we drill—they spend a lot of money—we are not certain that they are going to come up with a result. But, the value when they do is very high, so we want to do the same thing with R&D.”<sup>320</sup>

Kondos, an experienced metallurgist who had previously led research groups at Noranda and Inco, was hired in 2004 to help build a team of experienced metallurgists. This group focused on strategic challenges and became known as the Strategic Technology Solutions group. The most pressing challenge was the continued development of the company's flagship Goldstrike mine.

<sup>312</sup> Muir and Aylmore 2004, 541-543.

<sup>313</sup> Anon. 2015.

<sup>314</sup> Berezowsky and Sefton 1979, 2.

<sup>315</sup> Berezowsky and Sefton 1978, 6.

<sup>316</sup> See Marchbank et al 1996 and Thomas et al 1998.

<sup>317</sup> Kondos, Peter. (December 18, 2018) Personal Phone Interview. 16:00.

<sup>318</sup> Sabo 2014.

<sup>319</sup> Kondos, Peter. (December 18, 2018) Personal Phone Interview. 34:00.

<sup>320</sup> Kondos, Peter. (December 18, 2018) Personal Phone Interview. 3:00.

An early project involved the modification of the site's obsolete acid leach autoclaves. Much of the mine's remaining reserves were high in carbonate and could not be processed efficiently using an acid leach.<sup>321</sup> The team therefore developed an alkaline leach that permitted the facility to continue operation. However, the ores that could be processed in this system eventually ran out. The company faced the prospect of either abandoning its autoclave facilities or assigning them to a novel thiosulfate process. The establishment of the company's strategic R&D group, along with the acquisition of Placer Dome and its expertise in thiosulfate, made the latter option a possibility.

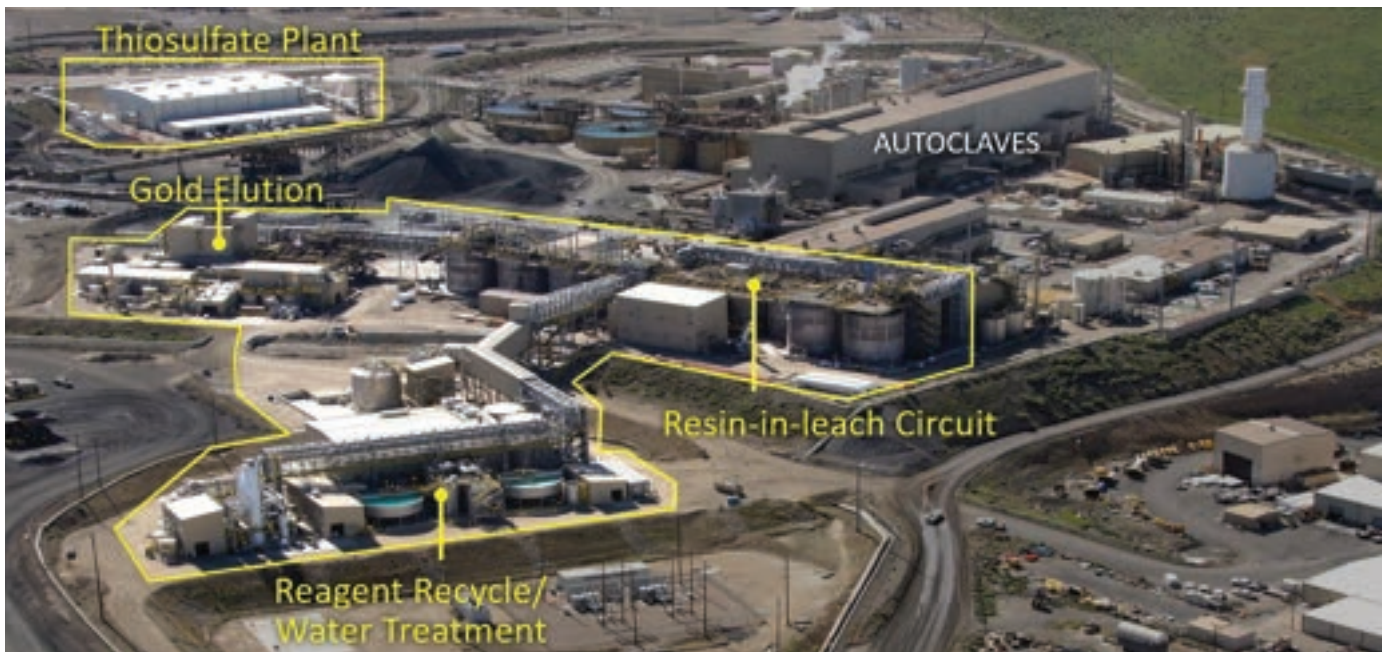
Implementing the thiosulfate process involved bridging the considerable gap between a challenging-but-well-researched chemical flowsheet into a functioning metallurgical facility. This process was done in partnership with a large number of research and industrial groups. Following the acquisition of Placer Dome, its research group in Vancouver became the Barrick Technology Centre and continued to work on the project. Lakefield Research of Peterborough, Ontario (now SGS Canada), also contributed to the development of the process.

Within six months of restarting work on the thiosulfate process at Barrick, it was decided to switch decisively to a calcium-based thiosulfate process over the more commonly researched ammonium thiosulfate. The plant that was eventually built to generate the calcium thiosulfate lixiviant

for the Goldstrike plant was constructed and operated as an integrated facility through an agreement with Tessenderlo Kerley of Phoenix, Arizona, a world leader in fertilizer production.<sup>322</sup>

In applying the process on an industrial scale, the research and development team at Barrick faced significant technical challenges. Among the most substantial was the problem of elution—the removal of the gold from an ion-exchange resin into a concentrated solution prior to the recovery of gold. The company sought the assistance of the Commonwealth Scientific and Industrial Research Organisation (CSIRO), an Australian government-funded laboratory that is a world leader in reagent use.<sup>323</sup> A second major bottleneck involved the engineering problem of rapidly recovering the pregnant thiosulfate solution into the ion-exchange resin before it could oxidize and lose its gold content.

A 16-month demonstration plant campaign was approved in 2010. Demolition of the plants' existing carbon-in-leach tanks began in 2012. Construction involved major upgrades to the existing leaching facility, as well as the construction of several new plants. The first gold from the new facility, known as the TCM (total carbonaceous matter) project, was poured in November 2014. The facility has a full capacity of 13,400 tonnes per day of ore. Project management and engineering was provided by Ausenco, an Australian company. The entire TCM project, which included upgrades to various parts of the plant, cost US\$620 million.<sup>324</sup>



**Figure 18:** The completed TCM facility at the Goldstrike mine (Image courtesy of Peter Kondos).

<sup>321</sup> Kondos, Peter. (December 18, 2018) Personal Phone Interview. 8:00.

<sup>322</sup> Kondos, Peter. (December 18, 2018) Personal Phone Interview. 18:40.

<sup>323</sup> Stokes 2015.

<sup>324</sup> Anon 2015.

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## THE BARRICK CALCIUM THIOSULFATE PROCESS

Many details of the TCM project have not been published. However, a general outline of the process can be given based on private interviews and media reports. Prior to entering the recovery process, the ore is processed in the existing pressure leach autoclaves where it is combined with water, oxygen, and limestone at 225°C. The oxidized ore slurry is contacted with thiosulfate in one of two banks of vertical stainless steel tanks, each 14.6 metres (48 feet) high. The gold thiosulfate particles are recovered using an ion-exchange resin consisting of fine beads. This is a commonly available industrial product used in various processes including water treatment.

The absorption of gold from the leach solution takes place within these tall tanks. Compared to cyanide, thiosulfate is a very unstable compound that oxidizes rapidly. The efficiency of Barrick's process comes from repeatedly contacting the thiosulfate with the slurry over successive stages in order to prevent anions of decomposed thiosulfate from bonding with the resin. When this process is complete, the pregnant resin is sent to an elution process where a concentrated gold solution is separated away from the resin using a chemical elutant. Both the resin and the elutant are recovered and recycled.

The concentrated gold solution is electrowon to produce cathode gold. This is smelted into doré bars for further refining. Thiosulfate reagent is recycled through a large reverse osmosis plant that recovers the vast majority of the reagent. The efficiency of this step has been critical to the project's viability. Researchers at Barrick have also patented a process for generating thiosulfate from the elemental sulphur developed during the pressure oxidation phase. By adding a reactant to the slurry, thiosulfate can be generated directly. Alternately, thiosulfate can be formed from sulphur dioxide produced during the roasting operation.<sup>325</sup> A future implementation of Barrick's thiosulfate process could incorporate some version of this technology.

## THE DECLINE OF RESEARCH CULTURE AT BARRICK

The development of Barrick's thiosulfate leaching process occurred at a time when several other major Canadian mining companies were paring back their in-house research departments. A period from the mid-1980s through the 2000s saw the industry largely turn away from the development of novel processing technologies. The decision by Barrick's leadership to consider scientific research as similar to prospecting—a roll of the dice with potentially large payouts for the sums involved—ran counter to this trend. The successful development and implementation of the thiosulfate process seems to have vindicated this decision.

This focus on research was not to last. The death of the company's founder, Peter Munk (1927–2018), followed by an acquisition of Randgold Resources, led to a change in leadership. As part of a broader effort aimed at restructuring around key assets and reducing a substantial debt load, the company has recently moved decisively away from its research and development focus. It has recently eliminated the executive position of chief innovation officer, and is seeking to sell its AuTec Innovative Extractive Solutions, a wholly owned subsidiary operating as its research and development division.<sup>326</sup>

The future of Barrick's thiosulfate leaching process remains uncertain. The company is reportedly willing to license the technology. Given the inevitable move to lower-grade ores, the process may see further applications. According to the process' developers, significant optimizations and improvements would follow from the experience gained in the technology's first implementation at the Goldstrike mine.<sup>327</sup>

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<sup>325</sup> Choi et al. 2009.

<sup>326</sup> MacDonald and McNish, 2018.

<sup>327</sup> Kondos, Peter. (December 18, 2018) Personal Phone Interview. 26:40.

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# CONCLUSION

Canada is an exporting country, with vast mineral resources spread across an equally vast landscape. The captive markets of its population centres are few and far between. Great efficiency is required to produce metal economically, since the cost of transporting products to market, or byproducts and intermediates for further processing, increases with distance.

The development of hydrometallurgy in Canada has been shaped by these circumstances of geography, climate, and settlement. For instance, the economic challenges surrounding the production of sulphuric acid, a byproduct of the smelting process, drove the development of pressure hydrometallurgy at Fort Saskatchewan, as well as its subsequent adoption at remote locations such as Trail, British Columbia; Flin Flon, Manitoba; and Voisey's Bay, Newfoundland.

Landscape and climate have shaped this technological pathway in other ways as well. In much of the world, heap leaching is a solution to processing marginal ore that favours the production of sulphuric acid through high-temperature operations. The Canadian climate is less amenable to this process. This consideration weighs in favour lower-temperature hydrometallurgical processes that produce elemental sulphur, which is more easily stockpiled or transported long distances to market.<sup>328</sup>

Inevitably, the move towards challenging ores, such as preg-robbing and refractory gold deposits, or arsenic containing ores, will favour the continued development of hydrometallurgical processes. Canada's longstanding status as a major mining country, and its record of achievement in the field of hydrometallurgy, suggest that Canadian mining companies have an important role to play. The completion of Vale's hydrometallurgical Long Harbour Processing Plant in 2014 provides reason for optimism.<sup>329</sup>

Yet, the move towards lower-grade ores also increases costs and reduces profits. This disincentivizes mining companies from continuing to invest in new and untried technologies.<sup>330</sup> Meanwhile, the ownership of most of Canada's large metallurgical companies has passed to foreign-owned multinationals that may have no particular interest in the long-term strategic development of Canada's mining sector. These, and other factors, have led to the closing and scaling back of many of the centres of research that developed the processes described in this report.<sup>331</sup>

In a 2011 survey of Canadian metallurgists, respondents selected words like "challenged" and "foreign" at over twice the rate of more positive assessments such as "dynamic" and "R&D leader" to describe the state of Canada's mining industry.<sup>332</sup> As this report was being written, Barrick Gold, once an outstanding example of a company committed to research and development, completed its merger with Randgold Resources, a company headquartered in the Channel Islands tax haven. Among the first casualties of this merger has been the company's Toronto-based research staff.<sup>333</sup>

If Canada is falling behind the technology curve in this sector, other countries continue to invest strategically in new metallurgical research. In several informal interviews conducted for this report, China, Sweden, Finland, and Australia were identified as having encouraged research and development in mining and metallurgy. Australia was repeatedly mentioned as outstanding in this field. The contribution to the development of Barrick's thiosulfate process by the Commonwealth Scientific and Industrial Research Organisation (CSIRO), a body funded by the Australian government, shows the benefit of this investment to the industry as a whole.<sup>334</sup>

When hydrometallurgy first emerged as a professional subdiscipline within the mining industry, there was great optimism for its efficacy and environmental benefits.<sup>335</sup> As the several case studies in this report indicate, some of this optimism was warranted, though numerous efforts were limited by technical challenges and the rapid, parallel evolution of pyrometallurgy. Nevertheless, the significance of this field extends well beyond the success or failure of a particular process. Chemical engineering touches all aspects of mining, from solvent extraction, to electrorefining, to the treatment of tailings. Continued research within the field of hydrometallurgy advances the mining industry as a whole.

During a 2016 interview with the Mining and Metallurgy Legacy Project, Canadian hydrometallurgist Gordon Ritcey reflected on a long career that began in the post-war years of uranium extraction, and lasted through the golden age of government-funded research and the boom in pressure hydrometallurgy. Like many Canadian metallurgists, he lamented the decline in research funding that, he believed, had taken place over the previous decade. Ritcey's reflections on his early career at the federal CANMET research laboratory serve as a reminder of the need to plan beyond the short term. He noted:

It was a time of freedom of thinking and doing... The criticism was that [the government labs had become] a

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<sup>328</sup> Kawulka et al. 1978. 133.

<sup>329</sup> Brent 2011.

<sup>330</sup> Mouat 2011, 22.

<sup>331</sup> Weidenhammer 2018, 61-62.

<sup>332</sup> Kapusta, Mackey, and Stubina 2011, 449.

<sup>333</sup> MacDonald and McNish, 2018.

<sup>334</sup> Stokes 2015.

<sup>335</sup> Wadsworth 1983, 6.

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glorified university... But... places like NRC, and the government labs at CANMET ...had proved that we had the talent to produce stuff for the future. It didn't have to be used today—it can be used in the future—and we should have that bank of information.<sup>336</sup>

There is little doubt that the renewal of this bank of innovative hydrometallurgical research would provide a lasting benefit to Canada's mining industry in a future of diminishing ore deposits and rising competition.

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<sup>336</sup> Gordon Ritcey, from "Gordon Ritcey—Full Interview," interviewed by William McRae, YouTube, February 29, 2016, video 43:00, <https://www.youtube.com/watch?v=M5Aj7VSmiDY>.

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