

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

CHINA CHENGDA ENGINEERING CO., LTD.
Petitioner

v.

DORIAMO LTD
Patent Owner

Case No. IPR2023-00365
Patent 8,591,852

DECLARATION OF GRAEME NORVAL

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Declaration of Graeme Norval

I. INTRODUCTION

1. I make this declaration based upon my own personal knowledge and, if called upon to testify, would testify competently to the matters contained herein.

2. I have been asked to provide technical assistance in the *inter partes* review of U.S. Patent No. 8,591,852 (“the ’852 Patent”). This declaration is a statement of my opinions on issues related to the unpatentability of Claims 1-10 of the ’852 Patent. I am being compensated at my normal rate, plus reimbursement for expenses, for my analysis. My compensation does not depend on the content of my opinions or the outcome of this proceeding.

II. EXPERIENCE AND QUALIFICATIONS

3. In formulating my opinions, I have relied upon my knowledge, training, and experience in the relevant art. My qualifications are stated more fully in my curriculum vitae, which has been provided as Appendix A. Here, I provide a brief summary of my qualifications.

4. Some highlighted information regarding my education, technical experience and publications is provided below.

5. I obtained my bachelor’s degree in chemical engineering in 1983, and subsequently completed both a research masters of Applied Science (1985) and a Ph.D. degree (1989) in Chemical Engineering from University of Toronto.

6. I began my work career in April 1989 with Canadian Industries Limited, which was a major Canadian chemical company. I worked as a research engineer, supporting the Sulphur Products business unit, which produced and marketed sulfuric acid and liquid sulfur dioxide, as well as the Forest Products business unit which produced and marketed chlorine, caustic soda, muriatic acid and hypochlorite bleach.

7. I continued in these roles through various corporate reorganizations and transitions until 2002. In June 2002, I opened my consulting business, GWN Chemical Consulting, Inc., and continued to work primarily in the inorganic process chemistry field, supporting small businesses primarily in Southern Ontario.

8. I began to teach chemical engineering at the University of Toronto in 2002, on a part-time basis, and became a full-time faculty member in 2007, until my retirement in 2021.

9. My area of expertise is in inorganic process chemistry, particularly that involving sulfur, chlorine and caustic soda chemistry. The treatment of brine for removal of impurities in the chlor-alkali process is identical to the treatment of brine in the Solvay process. The caustic soda produced in North American chlor-alkali competes with mined soda ash. In addition, the use of caustic soda in the pulp and paper industry involves the liming of a green liquor to give calcium carbonate, which is subsequently calcined to return the lime values.

10. I have worked with several companies to improve their operations and reduce both the caustic soda consumption and odor releases in the tail gas scrubbers. This chemistry involves the absorption of CO₂, as well as operation at a suitable solution pH. I developed and submitted the documentation packages to obtain Environmental Compliance Approvals for twelve small businesses which operate in the Province of Ontario. I have been on the Board of Directors of Organic Potash Corporation, which is a venture capital company working to produce potassium carbonate from cocoa pod husk ash, in west Africa. I believe that I have sufficient knowledge of soda ash production to offer this opinion.

11. My analyses set forth in this declaration are informed by my experience in the field of inorganic process chemistry. Based on my above-described experience, I am an expert in the field relevant to the '852 Patent at issue here and have been an expert in the relevant field since before the claimed priority date of the '852 Patent. I am familiar with how a person having ordinary skill in the art ("POSITA") would have understood and used the terminology found in the '852 Patent before and at the time of its filing.

III. LEGAL STANDARDS

12. My opinions are also informed by my understanding of the relevant law. I understand that the patentability analysis is conducted on a claim-by-claim and element-by-element basis, and that there are several possible reasons that a

patent claim may be found to be unpatentable.

13. I understand that earlier publications and patents may act to render a patent unpatentable for one of two reasons: (1) anticipation and (2) obviousness.

14. I understand that the prior art may render a patent claim “obvious.” I understand that two or more prior art references (e.g., prior art articles, patents, or publications) that each disclose fewer than all elements of a patent claim may nevertheless be combined to render a patent claim obvious if the combination of the prior art collectively discloses all elements of the claim and one of ordinary skill in the art at the time would have been motivated to combine the prior art in such a way. I understand that this motivation to combine need not be explicit in any of the prior art, but may be inferred from the knowledge of one of ordinary skill in the art at the time the patent was filed. I also understand that one of ordinary skill in the art is not an automaton, but is a person having ordinary creativity. I further understand that one or more prior art references, articles, patents or publications that disclose fewer than all of the elements of a patent claim may render a patent claim obvious if including the missing element would have been obvious to one of skill in the art (e.g., the missing element represents only an insubstantial difference over the prior art or a reconfiguration of a known system).

15. Under the doctrine of obviousness, a claim may be invalid if the differences between the invention and the prior art are such that the subject matter

as a whole would have been obvious at the time the invention was made to a POSITA to which the subject matter pertains.

16. To assess obviousness, I understand that I am to consider the scope and content of the prior art, the differences between the prior art and the claim, the level of ordinary skill in the art, and any secondary considerations to the extent they exist.

17. I understand that any evidence of secondary indicia of non-obviousness should be considered when evaluating whether a claimed invention would have been obvious to one of ordinary skill at the time of invention. These secondary indicia of non-obviousness may include, for example:

- a long felt but unmet need in the prior art that was satisfied by the claimed invention;
- commercial success of processes claimed by the patent;
- unexpected results achieved by the invention;
- praise of the invention by others skilled in the art;
- the taking of licenses under the patent by others; and
- deliberate copying of the invention.

18. I understand that there must be a nexus between any such secondary indicia and the claimed invention.

19. It is also my understanding that there are additional considerations that may be used as further guidance as to when the above factors will result in a finding

that a claim is obvious, including the following:

- the claimed subject matter is simply a combination of prior art elements according to known methods to yield predictable results;
- the claimed subject matter is a simple substitution of one known element for another to obtain predictable results;
- the claimed subject matter uses known techniques to improve similar devices or methods in the same way;
- the claimed subject matter applies a known technique to a known device or method that is ready for improvement to yield predictable results;
- the claimed subject matter would have been “obvious to try” choosing from a finite number of identified, predictable solutions, with a reasonable expectation of success;
- there is known work in one field of endeavor that may prompt variations of it for use in either the same field or a different one based on design incentives or other market forces if the variations would have been predictable to a POSITA;
- there existed at the time of conception and reduction to practice a known problem for which there was an obvious solution encompassed by the patent’s claims; and
- there is some teaching, suggestion, or motivation in the prior art that would have led a POSITA to modify the prior art reference or to combine prior art reference teachings to arrive at the claimed subject matter.

20. Finally, I understand that a claim may be deemed invalid for obviousness in light of a single prior art reference, without the need to combine references, if the elements of the claim that are not found in the reference can be supplied by the knowledge or common sense of one of ordinary skill in the relevant art.

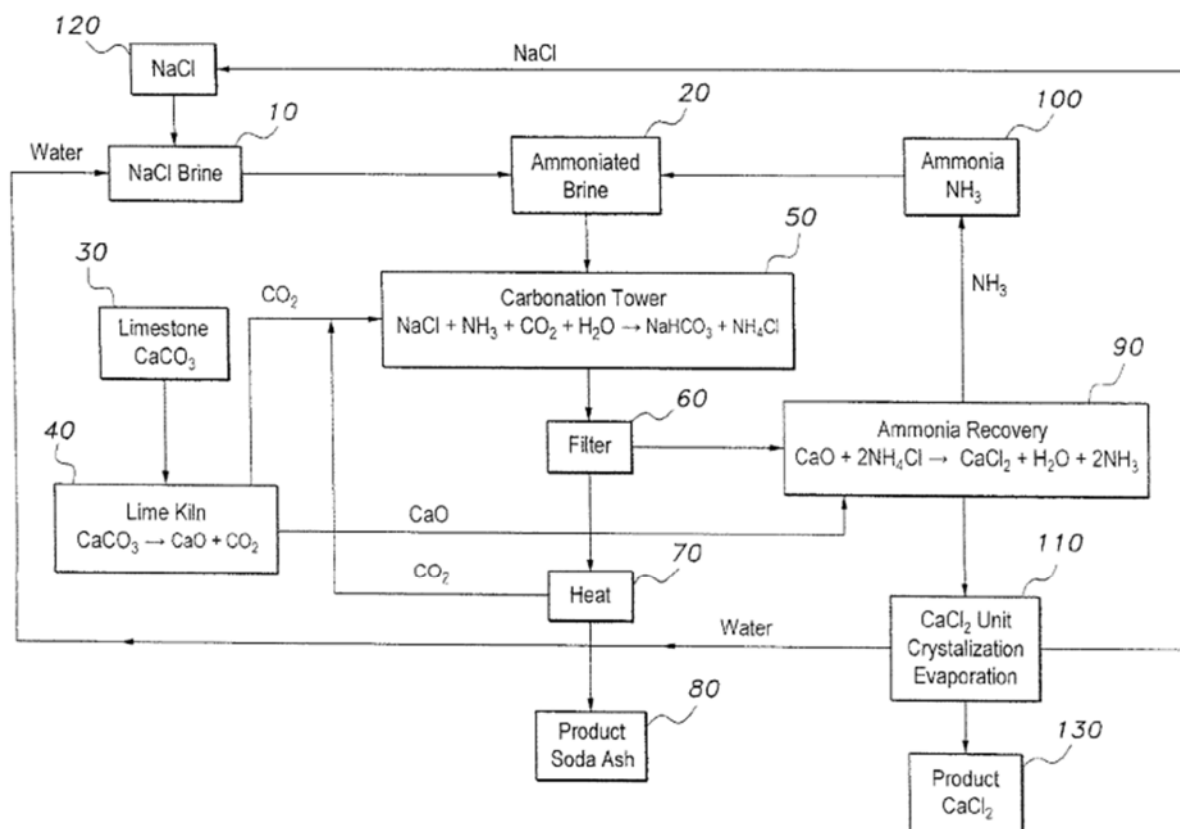
IV. OVERVIEW OF THE '852 PATENT

21. I have reviewed the '852 Patent, which is entitled "Method of producing soda ash and calcium chloride." EX1001. I understand that this patent was filed on October 15, 2012 and issued on November 26, 2013. *Id.* I also understand that this patent claims a priority date based on a parent application filed on February 22, 2012. *Id.*

A. Subject Matter of the '852 Patent

22. The '852 Patent involves a method of producing soda ash and calcium chloride. EX1001, 1:13-17. The '852 Patent explains in its "Background of the Invention" section that "[t]he most common method of producing soda ash (sodium carbonate) is the Solvay process. The Solvay process was developed into its modern form by Ernest Solvay during the 1860s." EX1001, 1:19-24. The '852 Patent allegedly provides "a modified version of the Solvay process that recycles ammonia, sodium chloride and water to eliminate harmful waste byproducts." *Id.*, 1:13-17; *see also id.*, 2:16-19 ("The method of producing soda ash and calcium chloride provides

an environmentally friendly method of producing soda ash and calcium chloride by recovering and recycling effluents and byproducts from a modified Solvay process.”). The sole drawing of the ’852 Patent summarizes the claimed processes:



EX1001, Figure.

23. “Detailed Description of the Preferred Embodiments” of the ’852 Patent consists of only six paragraphs in its entirety, which essentially restate the process flow depicted in the Figure of the ’852 Patent. *Id.* 2:16-3:7. While the ’852 Patent purports to teach “a modified version of the Solvay process that recycles ammonia, sodium chloride and water to eliminate harmful waste by products,” as I

will explain in further details below, methods of producing soda ash and calcium chloride with minimal waste, including those mentioned in the '852 Patent, were well known before the alleged invention of the '852 Patent.

B. Prosecution History of the '852 Patent

24. U.S. Patent Application No. 13/651,534 was filed on October 15, 2012, as a continuation of application No. 13/402,783 filed on February 22, 2012. EX1001. I understand that on April 15, 2013, following a first action interview, Applicant amended claims 1 and 11 to, among other things, recite additional steps in connection with waste recycling and calcium chloride production, and added claims 15-19. EX1009 ('852 File History), p.74-81. And in a June 12, 2013 office action, the Examiner rejected claims 1-10 under 35 U.S.C. §112(b) and §103. EX1009, p.90. The Applicant then further amended claims 1 and 11 (issued as claim 5) to recite using calcium oxide without slaking to overcome the Examiner's obviousness rejection. *Id.*, p.98-104. I understand that thereafter, the Examiner issued a Notice of Allowance. *Id.*, p.108. However, it is also my understanding that the Examiner did not consider the prior art relied upon herein for the grounds of invalidity. *See* EX1001, References Cited.

V. STATE OF THE ART AT THE TIME OF THE '852 PATENT

A. Introduction

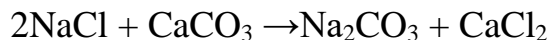
25. I provide the below technology overview for context, the subject matter

of which was well-known to persons of ordinary skill in the art before the time of the alleged invention.

26. Sodium carbonate, more commonly known as soda ash, is one of the largest volume inorganic chemicals produced globally. Sodium carbonate is widely used, for example, as a fluxing agent in glass manufacture, to manufacture sodium-based inorganic chemicals, or an alkali in soap and detergent applications. EX1010, p.11. The Solvay process has been used for its manufacture since the 1860s and continues to be used today globally. *Id.*, p.6.

27. Aspects of the Solvay process has been well documented in decades-old or even century-old publications. For example, the American Chemical Society arranged for the preparation and publication of a series of Monographs on the manufacture of chemicals beginning in 1919. As part of this series of publications, Dr. TePan Hou published a book entitled “The Manufacture of Soda” in 1923 (the second edition published in 1942). EX1013. Dr. Hou was known to be an expert on the manufacture of soda ash using the Solvay process having begun his work in 1919 at a Solvay plant in China. Certain of his work also appears in various books published in China. *See, e.g.*, EX1006.

28. The Solvay process for the production of soda ash may be summarized by the theoretical global equation involving the two main components: sodium chloride (NaCl) and calcium carbonate.



29. The commercial success of the Solvay process owes largely to the inexpensive sodium chloride and calcium carbonate (from limestone) supplies. EX1013, p.140. The Solvay process takes a series of process steps, beginning with a sodium chloride solution, which is commonly referred to as brine in the industry. EX1010, p.6. The brine absorbs carbon dioxide (CO₂), and a precipitate of sodium bicarbonate (NaHCO₃) is recovered. *Id.*, p.7. Because CO₂ is an acidic gas, the system pH needs to be controlled for the production of sodium bicarbonate precipitate. This is achieved through first buffering the brine solution with ammonia (NH₃), a weak base, to form ammoniated brine prior to CO₂ absorption. EX1010, p.6. The more expensive ammonia only acts as a medium (not consumed in the production process) and is continuously recycled. EX1003, p.51; *see also* EX1013, p.98 (“Ammonia is a comparatively expensive commodity.... It is now a systematic practice in ammonia soda plants to save all ammonia-bearing liquors and send them to the distiller for recovering NH₃.”). Solvay process is thus also referred to as ammonia soda process.

30. The sodium bicarbonate recovered from carbonation of the ammoniated brine is thermally treated to obtain the end product sodium carbonate (i.e., soda ash) and CO₂. The latter is routinely recycled to the carbonation process. EX1010, p.7.

31. The Solvay process adds lime or milk of lime to recover ammonia from

the process. Lime (CaO), sometimes referred to as quicklime, is obtained by calcination of limestone, which also produces CO_2 as another product to be used in the carbonation process. EX1010, p.8; EX1003, p.52. Hence, limestone is another key raw material besides salt for the Solvay process.

32. CaO reacts with water and forms Ca(OH)_2 , a base. The addition of dry lime or milk of lime consequently increases pH, drives the ammonia from the effluent, and leaves a solution of calcium chloride. The ammonia is then returned to the brine through absorption. EX1013, p.98.

33. The above ammonia-soda chemistry was resolved and equipment developed to exploit it in the 1860s. The basics of the process haven't changed since then.

B. Using Dry Lime Without Slaking for Ammonia Recovery

34. As I explained above, a base Ca(OH)_2 is added to recover ammonia from the effluent. Whether to add dry lime directly into the effluent, or to add milk of lime by first slaking the lime, are two options that both effectively lead to the same chemistry, and have been used in the field since the early days of the Solvay process. *See, e.g.*, EX1011 (U.S. Patent No 2,781,244), 4:11-25 (disclosing that the process of ammonia recovery can be adapted to function with solid lime); EX1012 (U.S. Patent No 2,781,245), 7:51-55 (“While the above specific examples relate to the use of milk of lime, the invention is not limited thereto, but includes the use of

sand-containing lime in other forms. Thus, instead of milk of lime, dry lime containing sand can be similarly used in the practice of the present invention.”). EX1010, p.8 (“In some operations, dry lime is used to produce a more concentrated calcium chloride by-product stream.”).

35. As another example, U.S. Patent No. 1,962,150 to Mohler et al. of the Solvay Process Company teaches the addition of dry lime for the recovery of ammonia and discusses extensively the advantages of using dry lime instead of milk of lime in this process:

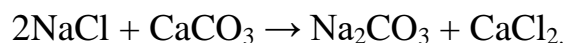
The advantages of the use of dry lime as hereinafter specified include ***the elimination of the step*** of producing milk of lime by the treatment of quick lime with water with a corresponding saving in the operative steps of the process. Again when dry lime is used, since the discharge still liquors are commonly concentrated to recover calcium chloride therefrom, ***a considerable saving in the cost of evaporating the discharge liquors*** results because of the absence of the water contained in the milk of lime. It will also be clear that ***the amount of steam and therefore fuel consumption required for operating the still is considerably decreased*** due to the decrease in the volume of liquor treated in consequence of avoiding the water in the milk of lime, and the use of less steam in turn results in less water of condensation being added to the discharge liquors and accordingly further decreases the cost of concentrating. There are other advantages attendant upon the use of dry lime, one of which is the ***increased capacity of the still when using dry lime*** instead of milk of lime.

EX1007, 2:84-106 (emphasis added); *see also id.*, 1:28-32 (“It has long been recognized that the use of dry quick lime as a reagent in the treatment of the

ammoniacal liquor to free the fixed ammonia, would prove the most economical method of using lime for this purpose[.]”). I further note that processes to use dry lime in Solvay processes have also been well-established. *See, e.g.*, EX1007, 2:118-4:25 (disclosing detailed means and structures for introducing dry lime into the process system); EX1005, [0040] (disclose means for introducing dry lime to the waste liquor to recover ammonia).

C. Waste Recycling

36. As discussed above, the overall chemistry of the Solvay process can be written as follows:



37. During the process, ammonia, carbon dioxide, and water also are used as reactants. A person of ordinary skill in the art has long recognized that for economic and environmental reasons, it is desirable to recycle these reactants as much as possible. For example, the Chinese patent CN 102,145,912 (“*Du*”) “guarantee[s] the recovery and recycling” of ammonia and carbon dioxide. EX1005, Abstract, [0011], [0020]. *See also* EX1004, p.19 (disclosing means for “comprehensive utilization and harmless treatment of distillation waste liquor and waste residue” including “recovery of CaCl_2 and NaCl ” and “using the purified wastewater to dissolve salt”).

38. As to sodium chloride and water recycling, it is well-known to recover

water by evaporation and recondensation, and to crystallize and separate sodium chloride from the waste solution. EX1013, p.125. For example, the water evaporation and crystallization of sodium chloride can be done in the process of calcium chloride production. EX1003, p.77-79; EX1008, p.35; EX1016, p.22 (“That is, solid sodium chloride should be precipitated first, and then the concentration of CaCl_2 in the liquid phase should reach 38% so as to meet the requirement for the commercial liquid calcium chloride component. To produce a molten or flaky solid product, the 38% CaCl_2 solution obtained is further concentrated and the edible salt precipitate once again precipitated is separated.”). POSITAs have long understood that sodium chloride solubility decreases as the concentration of the calcium chloride increases when water is removed through evaporation. EX1008, 403 (“The recovery of calcium chloride and salt from distillation waste liquor follows the phase diagram of CaCl_2 and NaCl binary system.”), Fig. 16-4 (showing decreasing solubility of NaCl with increasing CaCl_2 concentration):

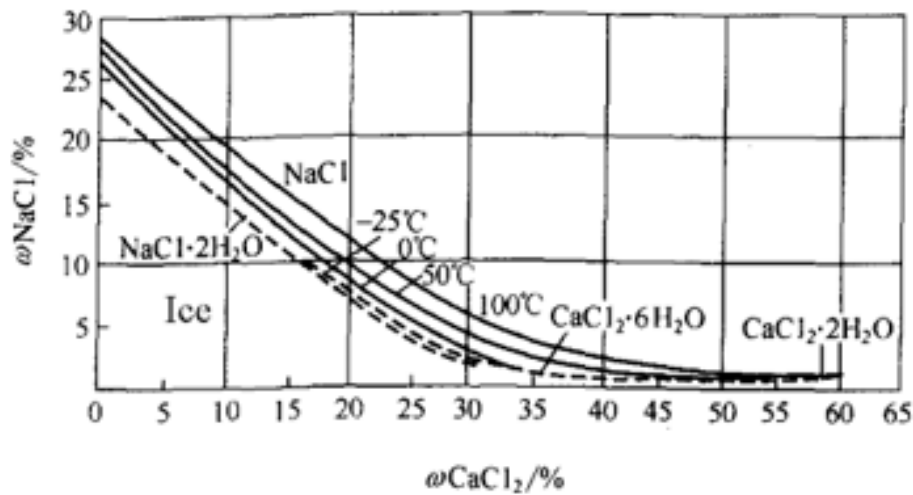


Fig. 16-4 Co-solubility of NaCl and CaCl₂ in water

39. Typical equipment used for evaporation is multiple effect evaporators, which produce a liquid water condensate; through condensing the water vapor, the heat content of the water vapor (steam) is reused. See EX1013, p.125 (“It is the aim of ammonia soda operators to keep the volume of distiller waste as small as possible... The clear solution containing calcium chloride, sodium chloride with a very small amount of dissolved calcium sulfate, is concentrated in a double-effect evaporator, when sodium chloride together with a small amount of calcium sulfate commences to crystallize out.”). Multiple effect evaporators are used in many industrial processes including the manufacture of caustic soda, the recovery of black liquid in pulp and paper, etc. Accordingly, it is my opinion that processes and equipment needed to recover various reactants are well known in the art.

VI. LEVEL OF ORDINARY SKILL IN THE ART

40. I am informed and understand that a POSITA is a hypothetical person who is presumed to know the relevant prior art. I understand that the actual inventor's skill is not determinative of the level of ordinary skill. I further understand that factors that may be considered in determining level of skill include: (i) type of problems encountered in art; (ii) prior art solutions to those problems; (iii) rapidity with which innovations are made; (iv) sophistication of the technology; and (v) educational level of active workers in the field. I understand that not all such factors may be present in every case, and one or more of them may predominate. In a given case, every factor may not be present, and one or more factors may predominate.

41. In my opinion, a POSITA at the time of invention of the '852 Patent would have had either (i) a Master of Science in Chemical Engineering, or an equivalent field, or (ii) a Bachelor of Science in Chemical Engineering or an equivalent field as well as two years of experience in the manufacturing or process development of inorganic alkali chemicals. Around the time of invention, I was familiar with this level of skill for inorganic alkali chemistry because I have a Ph.D. in Chemical Engineering and have worked and taught in the field for over thirty years.

VII. CLAIM CONSTRUCTION

42. I understand that "claim construction" is the process of determining a

patent claim's meaning. I also understand that, during an *inter partes* review, a claim construction analysis begins with the plain meaning of the claim term, which is the ordinary and customary meaning given to the term by those of ordinary skill in the art at the time of the invention. I also understand that the claims of a patent must be interpreted in light of the specification—that is, the claim language itself, the written description, and the figures in the patent—as well as the patent's prosecution history and/or any reexamination history.

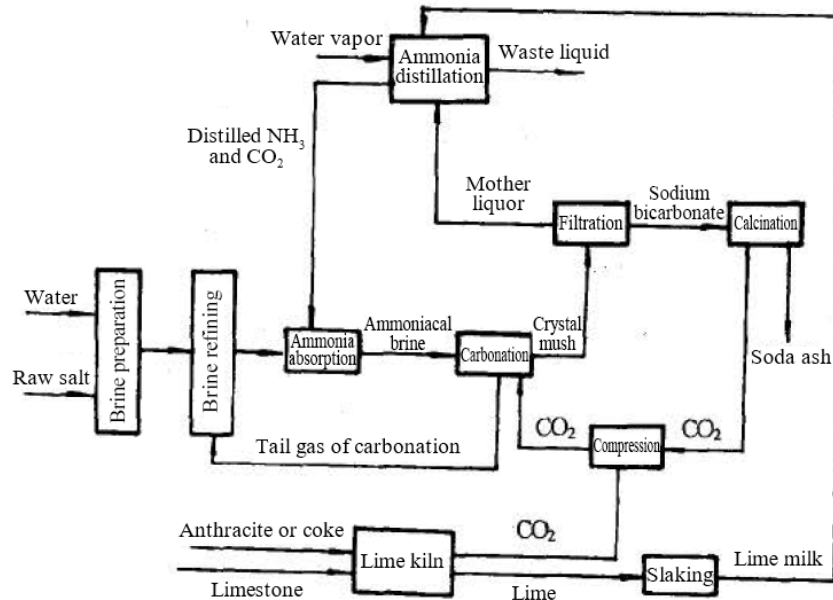
43. In my opinion, and for the purposes of this declaration, the claim terms of the '852 Patent do not require a construction. For purposes of this declaration, I interpret all claim terms in accordance with their ordinary and customary meaning, as would have been understood by a POSITA at the time of the alleged invention, having taken into consideration the language of the claims, the specification, and the prosecution history of record.

VIII. SUMMARY OF PRIOR ART

A. *Fu* (EX1003)

44. *Fu* was first published in September 1999. EX1003; EX1015(Hall-Ellis Decl), ¶¶44-50. *Fu* thus predates the '852 Patent by over ten years.

45. *Fu* describes a method of “Preparation of Soda Ash by ammonia soda Process.” EX1003, p.50. Figure 7-1 of *Fu* illustrates a flow diagram of the production process.



EX1003, Fig. 7-1.

46. *Fu* discloses that the process for producing soda ash also comprises processing clarified liquid to produce calcium chloride. *Id.* p.76 (“When the less-waste soda ash production complex is established, an efficient process for processing the clarified distillate can be promoted. The clarified liquid can be processed to produce calcium chloride or injected into oil wells after corresponding treatment.”). *Fu* recognizes that this production process may generate waste materials or harmful substances that are not environmentally friendly. EX1003, p.75-76. *Fu* sees the need to fully utilize natural resources and process the secondary materials resources. *Id.* (“When the less-waste process is established, the natural resources are *fully* utilized to ensure the demand for specified products, that is, the secondary material resources are processed, harmful wastes are not discharged into the atmosphere and water system, and heat loss to the surrounding environment should be minimized.”). To

that end, *Fu* teaches recycling the secondary material resources (waste residues) generated during the ammonia-soda process and turn them into useful products, such as calcium chloride, sodium chloride, ammonia, and brine solution. *Id.*

B. *Xi* (EX1004)

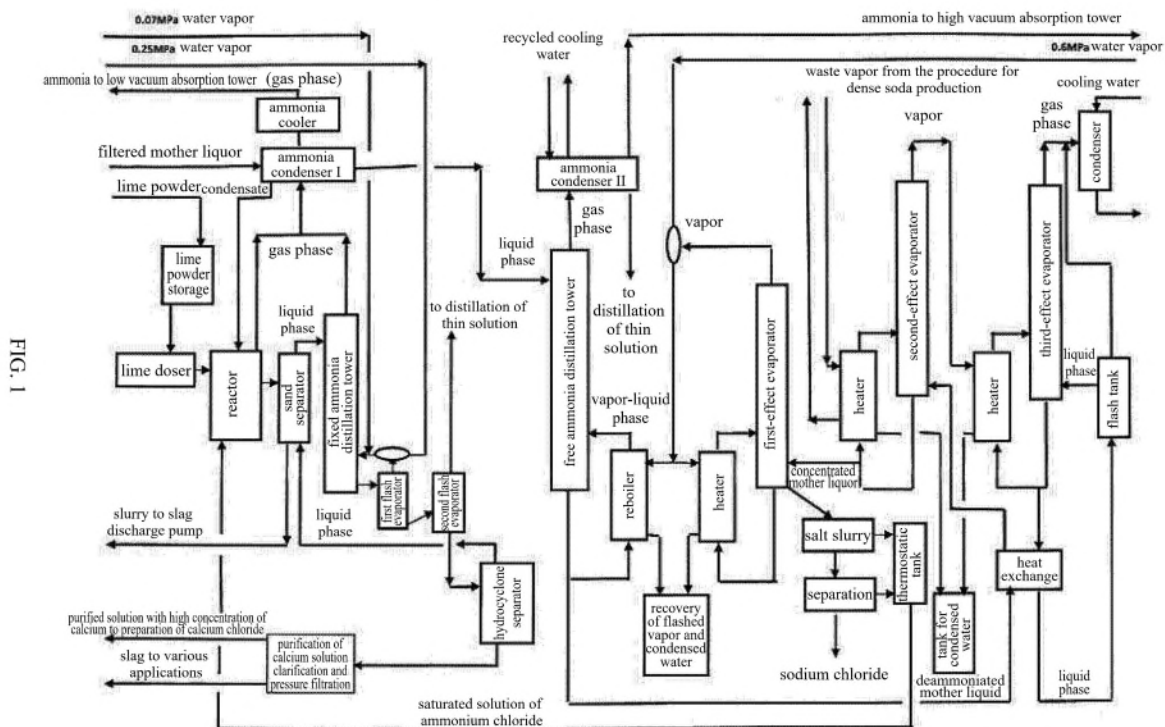
47. *Xi* was first published in 1990. EX1004; EX1015, ¶¶51-57. *Xi* thus predates the '852 Patent by over twenty years.

48. *Xi* discloses “comprehensive utilization and harmless treatment of distillation waste liquor and waste residue.” EX1004, p.19. *Xi* teaches a method of recovery of CaCl_2 and NaCl from distillation waste liquor after clarification and other potential use of the waste materials from the soda ash production process. *Id.* *Xi* also teaches methods to reduce water consumption, such as using dry quicklime to replace lime milk in ammonia distillation and recycling of wastewater. *Id.*

C. *Du* (EX1005)

49. *Du* (CN 102,145,912) was published on August 10, 2011. EX1005. *Du* thus predates the earliest possible priority date of the '852 Patent by about six months.

50. *Du* describes “[a] processing method for preparing calcium chloride solution using filtered mother liquor in ammonia-soda process.” EX1005, Abstract.



Id., Figure. *Du* teaches “the production of a calcium chloride solution through the reaction of the saturated ammonium chloride solution with lime powder in a reactor, and finally the purification of the calcium chloride solution, so as to obtain the finished product of calcium chloride solution.” *Id.*, Abstract. According to *Du*, the disclosed method can produce soda ash and calcium compound while guaranteeing the recovery and recycling of ammonia and carbon dioxide. *Id.*

51. Further, *Du* discloses that it aims to reduce liquid waste because it is “discharged in massive quantity, which is difficult to be handled completely.” *Id.*

¶[0002]. And, “for each ton of the product of soda produced by the ammonia-soda process, 10 cubic meters of waste liquid and slag need to be disposed. With such a high quantity, the waste liquid and slag not only cause pollution to the environment, but also lead to the waste of substances in the waste liquid, such as a certain amount of calcium chloride, sodium chloride, etc.” *Id.* ¶[0009].

D. *Hou* (EX1006)

52. *Hou* was published at least by 1990. EX1006; EX1015, ¶¶58-64. *Hou* thus predates the ’852 Patent by more than 20 years.

53. *Hou* discloses the production of “refined salt and calcium chloride” from the ammonia-soda process. EX1006, p.15. *Hou* emphasizes the importance of waste reduction: “in production using ammonia-soda process, it is hoped that the volume of waste liquor of distillation should be as small as possible.” *Id.* In addition, *Hou* describes the production of 75% calcium chloride, with a corresponding molecular formula of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$. *Id.* *Hou* then teaches the preparation of “anhydrous CaCl_2 ,” or 95% calcium chloride, by heating the 75% calcium chloride in a Furnace. *Id.*, p.16.

E. *Mohler* (EX1007)

54. *Mohler* was issued on June 12, 1934. EX1007. *Mohler* thus predates the ’852 Patent by more than 70 years.

55. *Mohler* discloses a method of recovery of ammonia from aqueous

ammonia liquors using dry lime. EX1007, title, 1:1-5. *Mohler* explains that an example of such an ammonia liquor is that obtained in the so-called Solvay or ammonia soda process. *Id.*, 1:6-8. *Mohler* also discloses that the use of dry quick lime as a reagent in the treatment of the ammoniacal liquor to free the fixed ammonia has been long recognized as an economical method. *Id.*, 1:28-32.

56. *Mohler* further explains the advantages of using dry quick lime in this process. *Id.*, 1:84-106. These advantages include: (1) the elimination of the step of producing milk of lime by the treatment of quick lime with water with a corresponding saving in the operative steps of the process; (2) a considerable saving in the cost of evaporating the discharge liquors results because of the absence of the water contained in the milk of lime; (3) the amount of steam and therefore fuel consumption required for operating the still is considerably decreased due to the decrease in the volume of liquor treated in consequence of avoiding the water in the milk of lime; (4) the use of less steam results in less water of condensation being added to the discharge liquors and accordingly further decreases the cost of concentrating; and 5) the increased capacity of the still when using dry lime instead of milk of lime. *Id.*

57. *Mohler* acknowledges that it is difficult to introduce solid lime into a reaction receptacle or container in a continuous and regulated manner and without the accompanying loss of gas or liquid from the system. *Id.*, 1:54-61. To overcome

these difficulties, *Mohler* teaches methods where dry lime is introduced into the system against pressure through a conduit in finely divided form. *Id.*, 2:1-9.

IX. SUMMARY OF PRIOR ART GROUNDS

Ground	Claims	Statutory Basis (35 U.S.C. §)	Prior Art
1A	1–10	103	<i>Fu in view of Xi</i>
1B	6-9	103	<i>Fu in view of Hou</i>
1C	10	103	<i>Fu in view of Xi and Hou</i>
1D	1–10	103	<i>Fu in view of Mohler</i>
1E	10	103	<i>Fu in view of Mohler and Hou</i>
2	1–10	103	<i>Du in view of Fu and Hou</i>

X. GROUND 1A: CLAIMS 1–10 ARE OBVIOUS OVER *FU* INVIEW OF *XI*

58. In my opinion, *Fu* in view of *Xi* renders Claims 1-10 obvious.

A. Motivation to Combine

59. *Fu* discloses that it is preferable to reduce waste “as much as possible” during the soda ash production process for the sake of environmental protection. EX1003, p.75-76. One of the main directions identified by *Fu* in establishing the less-waste process is “studying and developing a process system and a water circulation system to ensure that discharge of a liquid waste and harmful waste gas meets discharge standards of the local region.” *Id.*, p.76.

60. *Fu* discloses several examples as to how to reduce water consumption. *See, e.g., Id.* p.60 (“The crystals have good quality, that is, the particles are large and uniform, and easy to separate from mother liquor, thus the amount of washing water can be correspondingly reduced”); *Id.* p.65 (“However, if the temperature is too low, too much cooling water will be consumed, the tower is blocked and the operation cycle is shortened”) *Id.* p.79 (“The amount of condensed water generated in the evaporators is $5.5 \text{ m}^3/\text{t}_{\text{soda ash}}$ (calculated based on the production of 67% calcium chloride), and the condensed water is sent to a brine preparation field, and thus the water consumption can be appropriately reduced.”). Because *Fu* consistently teaches methods of generating less or no waste during the production of soda ash and calcium chloride, it is my opinion that it would have been obvious to a POSITA to combine teachings throughout *Fu* to achieve that goal.

61. Similarly, *Xi* discloses methods to utilize and treat distillation waste liquor and waste residue from the soda ash production process. EX1004, p.19. For example, *Xi* discloses reducing water consumption with “a water circulating system,” “[u]sing the purified wastewater to dissolve salt,” as well as “[u]sing dry quicklime to replace lime milk in ammonia distillation, and changing water cooling to air cooling to reduce water consumption.” *Id.*

62. In my opinion, a POSITA would have been motivated to combine the soda ash and calcium chloride production process of *Fu* with the teachings of *Xi*.

Both *Fu* and *Xi* are directed to the Solvay process and share the common objective of reducing waste production. More specifically, a main goal of *Fu* is to reduce waste generated in the production process, such as by recycling water and salt. EX1003, p.75-76. *Xi* Furthers that goal by using the dry quicklime without slaking to reduce water usage. EX1004, p.19. Indeed, these references are merely more explicit statements of the information already disclosed in Dr. Hou's earlier work, in which the volume of distillers waste is minimized, and that solid sodium chloride and calcium chloride can be produced. EX1013, p.125.

63. Therefore, with respect to *Fu*'s soda ash and calcium chloride production process, it is my opinion that a POSITA would have been motivated to incorporate *Xi*'s use of quicklime instead of milk of lime when reacting with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. Such a combination represents the use of a known technique (i.e., *Xi*'s use of quicklime in the soda ash production process) to improve a similar process (i.e., *Fu*'s soda ash production process) in the same way. Moreover, it is my opinion that a POSITA would have a reasonable expectation of success because replacing lime milk with quicklime does not materially change the chemistry involved in Solvay processes and leads to expected benefit of reduced water usage, as confirmed by *Xi*'s teachings. In addition, a POSITA would understand that this modification would not interfere with the other aspects of *Fu*'s processes because the dry lime,

upon contact with water in the waste liquor, will effectively turn into lime milk and perform the same functions as intended by *Fu*. Any equipment needed for such process adjustment would have been well-known in the art. *See, e.g.*, EX1007; EX1005. Indeed, the '852 Patent itself does not disclose any particular equipment needed for adding dry lime into the effluent to recover ammonia. *See* EX1001, 2:47-54.

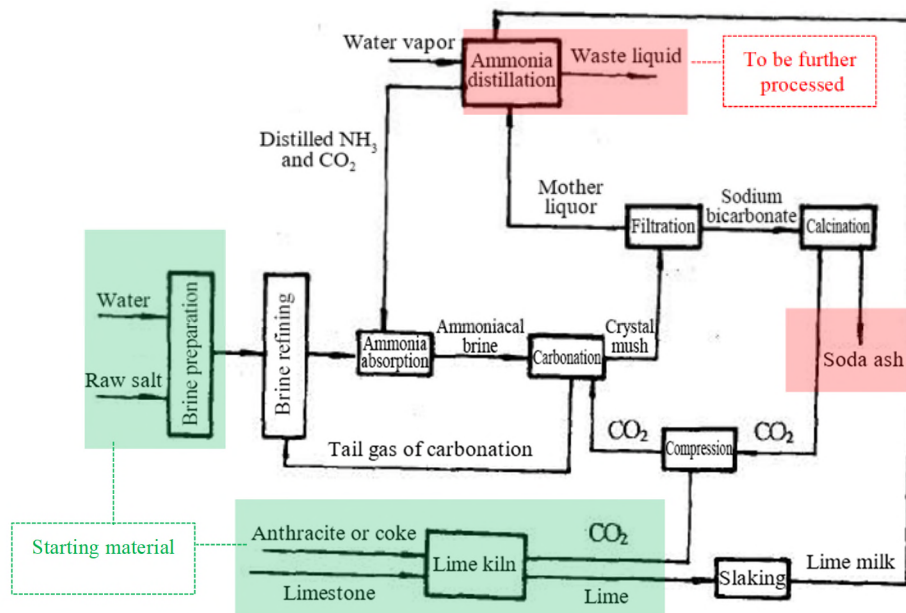
B. Claim 1

i.[1Pre] “A method of producing soda ash and calcium chloride, comprising the steps of:”

64. *Fu* discloses a method of producing soda ash (Na_2CO_3) and calcium chloride (CaCl_2). *Fu* discloses that “soda ash is prepared from edible salt (active ingredient NaCl) and limestone as follows:

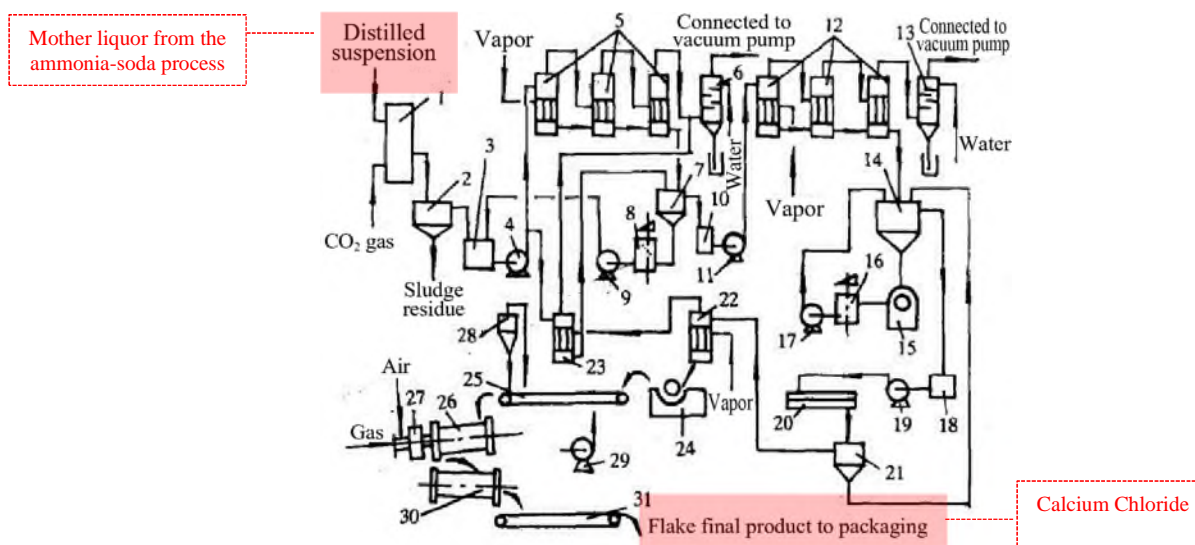


EX1003, p.50-51. Calcium chloride is shown above to be another product of the reaction. The flow diagram of Figure 7-1 in *Fu* also describes the production of soda ash via an ammonia-soda process (i.e., Solvay process).



EX1003, Figure 7-1 (annotated).

65. *Fu* further discloses that calcium chloride is produced from the waste liquid of the ammonia distillation tower as NaCl is simultaneously separated. EX1003, p.76-78. Figure 7-25 of *Fu* (titled “Production flow diagram of calcium chloride”) illustrates the process of producing calcium chloride:



EX1003, Figure 7-25 (annotated).

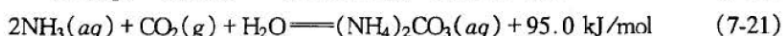
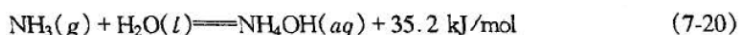
66. Accordingly, in my opinion *Fu* discloses claim 1[pre].

ii.[1a] “providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine;”

67. *Fu* discloses limitation [1a]. *Fu* discloses providing a source of brine. EX1003, p.51 (“***NaCl*** enters the production process in a form of an aqueous solution. ***Brine*** can be prepared from solid NaCl or natural brine can be used” and “The ***brine*** enters an ammonia absorption tower to absorb the required amount of NH₃.”) (emphasis added).

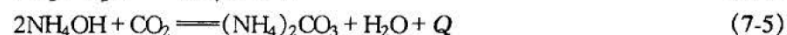
68. Similarly, *Fu* discloses a source of gaseous ammonia that ammoniates brine to form ammoniated brine:

Basic principle of ammonia absorption process: Ammonia absorption reaction and equilibrium partial pressure of gas above ammoniacal brine. The refined brine and the gas from ammonia distillation are mainly reacted as follows:

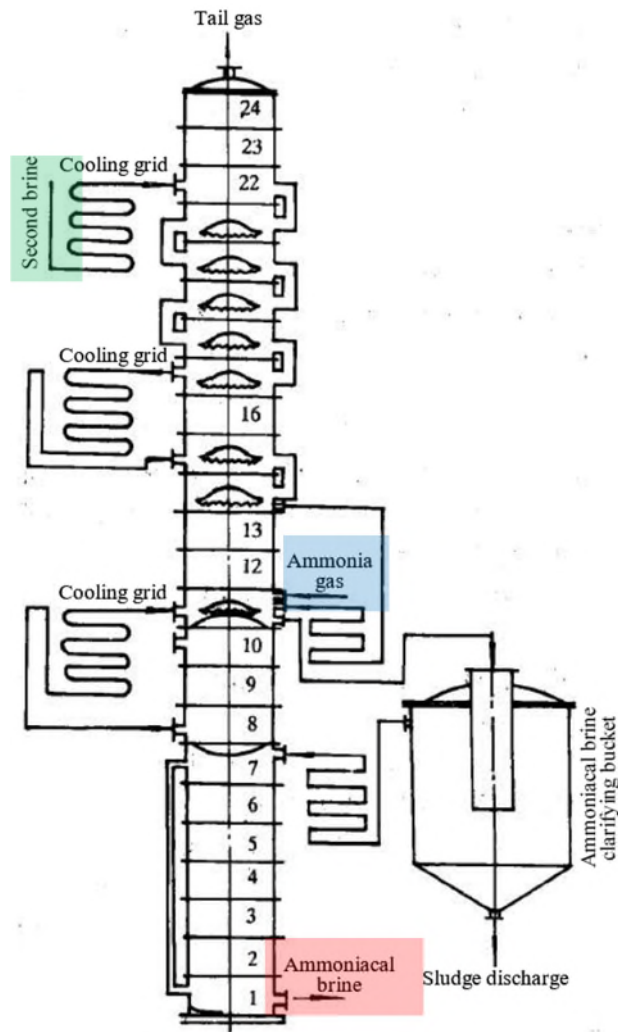


EX1003, p.57;

The brine enters an ammonia absorption tower to absorb the required amount of NH₃ and also absorbs a small amount of CO₂ at the same time.



EX1003, p.51. *Fu* also illustrates the ammonia absorption tower—a main device for ammonia absorption in Figure 7-7:



EX1003, Figure 7-7 (showing the production of ammoniated brine with gaseous ammonia using an ammonia absorption tower).

iii. [1b] “heating limestone to produce calcium oxide and carbon dioxide;”

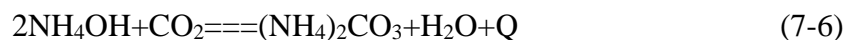
69. *Fu* discloses limitation [1b]. *Fu* discloses heating limestone to produce calcium oxide and carbon dioxide. EX1003, p.52 (“The carbonation process requires a large amount of CO₂ gas, such that limestone is calcined to produce CO₂ and lime.”), p.53 (“Practice has proved that the temperature of burning limestone is

generally controlled within a range of 940-1200°C.”). A POSITA would have understood that lime and calcium oxide refer to the same thing.

iv.[1c] “reacting the ammoniated brine with the carbon dioxide produced by the step of heating the limestone to produce sodium bicarbonate, ammonium chloride and a brine effluent;”

70. *Fu* discloses limitation [1c]. *Fu* discloses that the ammoniated brine reacts with carbon dioxide to produce sodium bicarbonate and ammonium chloride:

Ammoniacal brine with a low carbonation degree is cooled and then subjected to a carbonation process, and enters a tower here, and CO₂ is introduced.



EX1003, p.51-52. After the carbonation process and precipitation of sodium bicarbonate, a brine effluent (i.e., the salt-containing liquid waste) is thereafter produced as well. EX1003, p.52 (“[i]n an actual production, the precipitation amount of NaHCO₃ is equivalent to 70%-75% of the amount of Na brought in by NaCl, and the remaining Na remains in the solution and cannot be used in a subsequent process.”).

71. In addition, *Fu* discloses that at least some of the carbon dioxide reacting with the ammoniated brine is produced by the step of heating the limestone. EX1003, p.52 (“The gas from calcination, and absorption and washing contains

approximately less than half the amount of CO₂ required to form NaHCO₃. The remaining CO₂ comes from a lime kiln. Calcium carbonate is decomposed into CO₂ and CaO in the lime kiln.”).

v.[1d] “calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide;”

72. *Fu* discloses limitation [1d]. *Fu* teaches that “A filter cake containing NaHCO₃ is calcined to obtain final products Na₂CO₃ and CO₂.” EX1003, p.52.

vi.[1e] “reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia; wherein said step of reacting the calcium oxide with the ammonium chloride and brine effluent comprises reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone”

73. *Fu* alone or further in view of *Xi* renders limitation [1e] obvious. As discussed above regarding [1c], *Fu* discloses reacting ammoniated brine with carbon dioxide to produce sodium bicarbonate, ammonium chloride and a brine effluent. *See* Section X.B.iv. *Fu* further discloses producing calcium chloride, brine effluent, and ammonia by reacting calcium hydroxide with ammonium chloride and brine effluent. EX1003, p.52 (“A large amount of NH₃ remains in the mother liquor in a form of NH₄Cl, NH₄HCO₃ and (NH₄)₂CO₃. The NH₃ in the mother liquor must be distilled out and returned for absorption. The ammonia is driven out by steam in a gas-liquid counterflow device. ... The reaction is as follows: 2NH₄Cl+Ca(OH)₂===2NH₃+CaCl₂+2H₂O (7-12).”).

74. To the extent that *Fu* does not expressly disclose “reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone,” it is my opinion that a POSITA would have found it obvious to do so. *Fu* discloses heating limestone to produce calcium oxide and carbon dioxide. EX1003, p.52 (“Calcium carbonate is decomposed into CO₂ and CaO in the lime kiln.”), p.53 (“Practice has proved that the temperature of burning limestone is generally controlled within a range of 940-1200°C.”). In my opinion, a POSITA would have understood that when added to the ammonium chloride and brine effluent, the calcium oxide will react with water and turn into calcium hydroxide (a base) before reacting with ammonium chloride (a weak acid) to drive out ammonia. Whether dry calcium oxide is added to the liquid effluent with or without first slaking the calcium oxide, the chemical reactions remain the same. It is my opinion that a POSITA would understand that this modification would not interfere with the other aspects of *Fu*’s processes.

75. In addition, in my opinion a POSITA would understand that slaking the calcium oxide requires a separate step of adding water to the dry calcium oxide. It is my opinion that it would have been an obvious variation for a POSITA to use the calcium oxide produced by heating the limestone and add it directly to the ammonium chloride and brine effluent instead of carrying out a separate step to first slake the calcium oxide with water. *See, e.g.*, EX1011, 4:11-25 (disclosing that the

process of ammonia recovery can be adapted to function with solid lime); EX1012, 7:51-55 (“While the above specific examples relate to the use of milk of lime, the invention is not limited thereto, but includes the use of sand-containing lime in other forms. Thus, instead of milk of lime, dry lime containing sand can be similarly used in the practice of the present invention.”); EX1010, p.8 (“In some operations, dry lime is used to produce a more concentrated calcium chloride by-product stream.”). Moreover, in my opinion a POSITA would have been motivated to add calcium oxide directly to the ammonium chloride and brine effluent without introducing additional water, because it is desirable to reduce water consumption and cost for recycling the added water, particularly in view of *Fu*’s emphasis of waste reduction.

76. Alternatively, as described in Section X.A, it is my opinion that a POSITA would have been motivated to follow *Xi*’s express teaching of “using the dry quicklime” to “replace lime milk in ammonia distillation” of *Fu*’s method of recycling ammonia and producing calcium chloride. EX1004, p.19. Given that both *Fu* and *Xi* aim to reduce waste during the production process, in my opinion a POSITA would understand that, in the combination of *Fu* and *Xi*, the process will consume less water when quicklime is used instead of slaked lime.

77. Accordingly, it is my opinion that it would have been obvious for a POSITA to combine *Fu* and *Xi* and directly react the mother liquor with quicklime (calcium oxide) to produce calcium chloride, brine effluent and ammonia without

slaking the calcium oxide in an effort to reduce water consumption in the soda ash production process. Therefore, in my opinion *Fu* alone in view of the POSITA's knowledge, or *Fu* in view of *Xi*, renders obvious claim 1[e].

vii.[1f] “recycling the ammonia to be used in the step of ammoniating the brine; and”

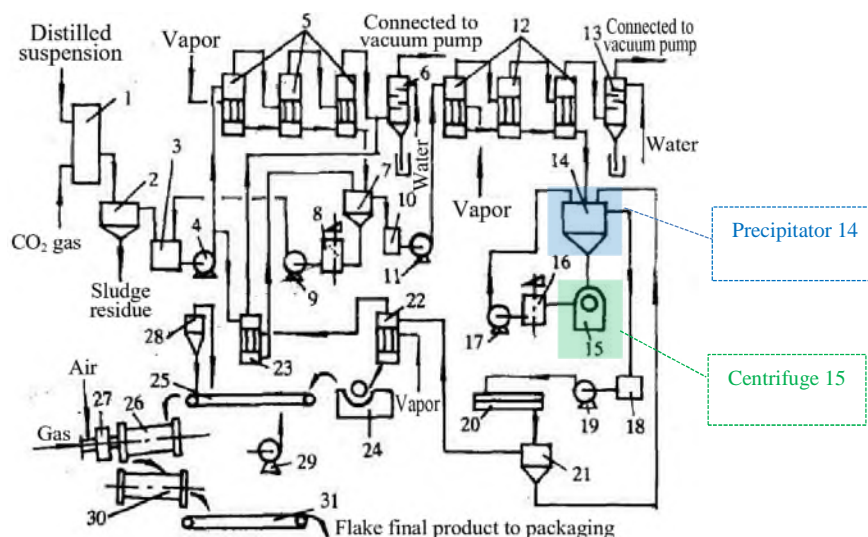
78. *Fu* discloses limitation [1f]. *Fu* teaches that the ammonia is continuously recycled because it is “more expensive” and “only acts as a medium here.” EX1003, p.51. To achieve that goal, “[t]he NH₃ in the mother liquor must be distilled out and returned for absorption.” *Id.*, p.52. *Fu* further discloses when the ammonia that comes from the ammonia distillation tower is recycled, “it is used for ammonia absorption [.]” *Id.*, p.57.

viii.[1g] “recycling the water from the brine effluent to be used in the step of ammoniating the brine by crystallizing sodium chloride from the mixture of calcium chloride and brine effluent, separating the crystallized sodium chloride from the effluent by centrifuging the effluent, and recycling the crystallized sodium chloride;”

79. *Fu* discloses limitation [1g]. *Fu* discloses that sodium chloride is crystalized and separated from the mixture of calcium chloride and brine effluent by concentrating the liquid through evaporation. EX1003, p.77-79 (“A method for preparing calcium chloride from the waste liquid of the ammonia distillation tower and simultaneously separating NaCl is introduced below.... The clarified distillate containing the seed crystal is ... concentrated by evaporation to contain 18% CaCl₂.”)

... The partially concentrated and deseeded distillate is ... concentrated to contain 38% calcium chloride and a main part of sodium chloride is separated.”). A POSITA would have understood that NaCl crystallizes and precipitates from the solution when the liquid is concentrated through evaporation.

80. *Fu* discloses that the crystallized sodium chloride is separated from the effluent by centrifuging the effluent and recycled for soda ash production. *Id.*, p.76 (“NaCl is returned to the production process for preparing soda ash”), p.79 (“The sodium chloride suspension in the 38% calcium chloride solution flows into a precipitator 14, a thick part then enters a centrifuge 15 from the precipitator, and solid sodium chloride is sent to the user or returned to soda ash production”); *id.* Figure 7-25 (showing a process diagram of preparing calcium chloride and simultaneously separating NaCl):



81. *Fu* further teaches recycling water from the brine effluent by

evaporation and condensation to be used in the step of ammoniating the brine¹. *Id.* p.79 (“The amount of condensed water generated in the evaporators is 5.5 m³/t_{soda ash} (calculated based on the production of 67% calcium chloride), and the condensed water is sent to a brine preparation field, and thus the water consumption can be appropriately reduced.”).

ix.[1h] “evaporating any water remaining to produce water vapor;”

82. *Fu* discloses limitation [1h]. *Fu* discloses that “[t]he clarified 40% calcium chloride solution is sent to an evaporator 22 at a normal pressure, where the solution is concentrated to a molten state and contains 72% CaCl₂” and forming “flakes containing the main substance of about 78% CaCl₂.” EX1003, p.79. In my opinion, a POSITA would understand when the solution is “concentrated,” water is evaporated from the solution in the evaporator. It is also my opinion that a POSITA would understand it is not practically possible, nor necessary, to evaporate *all* water

¹ In my opinion, a POSITA would have understood, in view of the specification, that the step of ammoniating the brine includes the brine preparation step. Indeed, the ’852 Patent only mentions a single purpose of the recycled water, that is to “make more brine.” EX1001, 2:64-66. Accordingly, “recycling the water from the brine effluent to be used in the step of ammoniating the brine” refers to the use of recycled water in brine preparation.

remaining. Indeed, element 1[n] recites “forming an approximately 77 wt % solution of calcium chloride flakes,” which is calcium chloride dihydrate known to contain water. EX1001, claim 1; *see also id.*, 2:66-67 (“Any remaining water is evaporated to leave the end product of calcium chloride (CaCl₂)[.]”) Accordingly, in my opinion, a POSITA reading the ’852 Patent would understand that “any water” only refers to the amount of water that needs to be evaporated to arrive at the end calcium chloride products and does not require removal of “all” water.

x.[1i] “recovering the produced calcium chloride;”

83. *Fu* discloses limitation [1i]. *Fu* teaches that the produced calcium chloride is recovered. EX1003, p.79 (“The hot flakes containing the main substance of 78% CaCl₂ are discharged into a cooling rotary cylinder ... The cooled final product flakes are conveyed by a conveyor for packaging and then transported into a final product warehouse.”).

xi.[1j] “recycling the water vapor and condensing into water;”

84. *Fu* discloses limitation [1j]. According to *Fu*, water vapor is generated in the evaporators and recycled. EX1003, p.79 (“The amount of condensed water generated in the evaporators is 5.5 m³/t_{soda ash} (calculated based on the production of 67% calcium chloride), and the condensed water is sent to a brine preparation field, and thus the water consumption can be appropriately reduced.”). *Fu*’s process flow diagram of calcium chloride, illustrated in Figure 7-25, includes a number of

evaporators such as 5, 12, and 22. *Id.*, Figure 7-25. For example, *Fu* discloses that “[t]he clarified 40% calcium chloride solution is sent to an evaporator 22 at a normal pressure, where the solution is concentrated to a molten state and contains 72% CaCl_2 .” *Id.* p.79. *Fu* explains that the amount of generated condensed water, $5.5 \text{ m}^3/\text{t}_{\text{soda ash}}$, is calculated based on the production of 67% calcium chloride. *Id.* Therefore, the condensed water that is “sent to a brine preparation field,” i.e., recovered, contains water generated from all evaporators, including evaporator 22.

xii.[1k] “wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method;”

85. *Fu* discloses or renders obvious limitation [1k]. *Fu* discloses that the crystalized sodium chloride is used for the providing of fresh brine. EX1003, p.76 (“solid sodium chloride is sent to the user or returned to soda ash production.”). In addition, *Fu* discloses that “the condensed water is sent to a brine preparation field, and thus the water consumption can be appropriately reduced.” *Id.* p.79.

86. It is my opinion that a POSITA would have understood from the disclosures of *Fu* that the crystalized sodium chloride and condensed water are combined for the providing of fresh brine because both the sodium chloride and the condensed water were sent to a brine preparation field and returned to soda ash production.

87. Alternatively, in my opinion it would have been obvious for a POSITA

to mix the recycled sodium chloride and condensed water of *Fu* to provide the fresh brine to be further used in soda ash production.

xiii.[11] “collecting the produced calcium chloride;”

88. *Fu* discloses limitation [11]. *Fu* teaches that “[t]he hot flakes containing the main substance of 78% CaCl_2 are discharged into a cooling rotary cylinder ... The cooled final product flakes are conveyed by a conveyor for packaging and then transported into a final product warehouse.” EX1003, p.79.

xiv.[1m] “separating residual salt; and”

89. *Fu* discloses limitation [1m]. As discussed above regarding limitation [1g], *Fu* discloses that “[t]he partially concentrated and deseeded distillate is ... concentrated to contain 38% calcium chloride and *a main part* of sodium chloride is separated.” EX1003, p.77-79 (emphasis added).

90. *Fu* further discloses that residual salt is separated from the calcium chloride solution, when “the solution is further concentrated.” EX1003, p.79 (“In the vacuum crystallizer 20, the solution is further *concentrated* to contain 40% CaCl_2 and at this time, the *remaining* amount of sodium chloride is precipitated into a solid phase”) (emphasis added); *see also id.* (“In some production processes of calcium chloride, the vacuum crystallizer is replaced by an evaporator group, that is, the evaporators are used to concentrate CaCl_2 to 48%, and then the sodium chloride is separated.”).

91. Alternatively, in my opinion it would have been obvious for a POSITA to separate residual salt from the calcium chloride product as a POSITA would have understood that to be an effective way to ensure the quality of the calcium chloride produced. *See, e.g.*, EX1008, p.36 (“[t]he technological process is characterized by fully removing calcium (CaCl_2) in salt making and fully removing salt during calcium chloride making to ensure the quality of both products.”); EX1016, p.22 (“That is, solid sodium chloride should be precipitated first, and then the concentration of CaCl_2 in the liquid phase should reach 38% so as to meet the requirement for the commercial liquid calcium chloride component. To produce a molten or flaky solid product, the 38% CaCl_2 solution obtained is further concentrated and the edible salt precipitate once again precipitated is separated.”).

xv.[1n] “forming an approximately 77 wt % solution of calcium chloride flakes.”

92. *Fu* discloses limitation [1n]. *Fu* discloses forming calcium chloride flakes with approximately 77 wt% calcium chloride. EX1003, p.79 (“The hot flakes containing the main substance of 78% CaCl_2 are discharged into a cooling rotary cylinder.”). It is my opinion that a POSITA would understand that calcium chloride flakes containing 78% CaCl_2 satisfies the requirement of “approximately 77 wt % solution of calcium chloride flakes.” Further, it is my opinion that a POSITA would have known that a common form of calcium chloride hydrates is $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, in which the weight percentage of calcium chloride is 75.5%. *See, e.g.*, EX1014, p.4

(disclosing calcium chloride products in “flake form” that contains “approximately 78% calcium chloride”). Thus, in my opinion it would also have been obvious to a POSITA to form an approximately 77 wt % solution of calcium chloride flakes.

C. Claim 2

- i. **“The method of producing soda ash and calcium chloride as recited in claim 1, further comprising the step of filtering the mixture of sodium bicarbonate, ammonium chloride, and brine prior to said calcining step, the brine and soluble ammonium chloride being removed for recovery of sodium chloride and ammonia.”**

93. *Fu* in view of *Xi* renders obvious claim 2. Further to the discussion above regarding Claim 1, *Fu* discloses the step of filtering the mixture of sodium bicarbonate, ammonium chloride, and brine prior to the calcining step and the recovery of ammonia from the mother liquor:

In the production of soda ash, the filtration process is used to *separate NaHCO₃ crystals* in a suspension from the carbonation tower. The obtained crystals are sent to a calcination section, and mother liquor together with washing water (called filtrate) is sent to the ammonia distillation section to *recover ammonia*.

EX1003, p.65 (emphasis added).

94. *Fu* also discloses recovering sodium chloride from the mother liquor. *See, e.g., id.*, p.79 (“The sodium chloride suspension in the 38% calcium chloride solution flows into a precipitator 14, a thick part then enters a centrifuge 15 from the precipitator, and solid sodium chloride is sent to the user or returned to soda ash production.”).

D. Claim 3

- i. **“The method of producing soda ash and calcium chloride as recited in claim 2, wherein said step of calcining the sodium bicarbonate comprises heating the filtered sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate.”**

95. *Fu* in view of *Xi* renders obvious claim 3. Further to the discussion above regarding claims 1 and 2, *Fu* teaches that “[s]odium bicarbonate is an unstable compound, and can be decomposed at a room temperature and rapidly decomposed by increasing the temperature. ... when the temperature is 100-101°C, the decomposition pressure has reached 0.1 MPa, that is, NaHCO₃ can be completely decomposed. ...in fact, the soda ash discharging temperature of the calcination is mostly controlled at 165-190°C.” EX1003, p.67.

E. Claim 4

- i. **“The method of producing soda ash and calcium chloride as recited in claim 1, further comprising the steps of condensing the water vapor and recycling the condensed water vapor for the formation of fresh brine with the recycled sodium chloride.”**

96. As discussed above regarding limitation [1k], *Fu* discloses that the recycled sodium chloride is used for the providing of fresh brine by combining with the condensed water vapor. At least for the same reasons discussed in Section X.B.xii, *Fu* in view of *Xi* renders obvious claim 4.

F. Claim 5

i. [5Pre] “A method of producing soda ash and calcium chloride, comprising the steps of:”

97. *Fu* disclosed a method of producing soda ash and calcium chloride for the same reasons above regarding [1Pre]. *See* Section X.B.i.

ii. [5a] “mixing sodium chloride and water to form fresh brine; providing ammonia gas; bubbling the ammonia gas through the fresh brine to form ammoniated brine;”

98. *Fu* discloses limitation [5a]. *Fu* discloses that brine is “prepared from solid NaCl” “in a form of an aqueous solution.” EX1003, p.51. *Fu* also discloses that “[t]he brine enters an ammonia absorption tower to absorb the required amount of NH₃” and “[t]he ammonia absorption tower is a multi-section cast iron single bubble-cap tower.” *Id.* p.51, p.59. In my opinion, a POSITA would have understood that gas absorption is performed by bubbling gas through liquid and a multi-section cast iron bubble-cap tower, which is a typical bubbler used in the industry to enhance gas-liquid exchange. *See* EX1003, p.63-64 (disclosing a “hat-shaped plate,” i.e., a “bubble cap” “arranged for dispersing air bubbles on the edges of the plates and the hats to increase the contact area of gas and liquid to promote absorption.”); *see also* EX1016, 20 (“The gas comes up from the lower column ring of the ammonia absorption equipment, bubbles after passing through the brine layer on the column tray, and enters the other column ring of the ammonia absorber or scrubber.”).

iii. [5b] “heating limestone to produce dry calcium oxide and carbon dioxide;”

99. *Fu* discloses limitation [5b] for the same reasons above regarding [1b].

See Section X.B.iii.

iv. [5c] “bubbling the carbon dioxide through the ammoniated brine in a carbonation tower to produce sodium bicarbonate, ammonium chloride, and a brine effluent, the sodium bicarbonate precipitating in the brine effluent and the ammonium chloride being soluble in the brine effluent;”

100. *Fu* discloses limitation [5c]. *Fu* discloses that “[a]mmoniacal brine with a low carbonation degree is cooled and then subjected to a carbonation process, and enters a tower here, and CO₂ is introduced.” EX1003, p.51. In addition, “[t]he upper part of the tower is a sieve plate and a bubble cap.” *Id.*, p.64. A POSITA would have understood that when the carbon dioxide is introduced into the carbonation tower, it is bubbling through the ammoniated brine facilitated by the disclosed tower structure. *See* EX1003, p.63 (“Teeth are arranged for dispersing air bubbles on the edges of the plates and the hats to increase the contact area of gas and liquid to promote absorption.”).

101. *Fu* discloses the limitation “to produce sodium bicarbonate, ammonium chloride, and a brine effluent” for the same reasons above regarding [1c]. Section X.B.iv.

102. *Fu* further discloses “the sodium bicarbonate precipitating in the brine

effluent and the ammonium chloride being soluble in the brine effluent.” EX1003, p.51 (“ NaHCO_3 is seldom dissolved in mother liquor of soda ash preparation, such that it is precipitated in a form of crystals.”). In addition, it is my opinion that it is well-known, and a POSITA would have understood, that the ammonium chloride produced during the carbonization process is soluble in the brine effluent.

v. [5d] “filtering the brine effluent to remove the precipitated sodium bicarbonate”

103. *Fu* discloses limitation [5d]. *Fu* discloses that “[t]he NaHCO_3 crystals suspended in taking-out liquid of the carbonation tower are subjected to a solid-liquid separation by a rotary vacuum filter.” EX1003, p.52.

vi. [5e] “calcining the filtered sodium bicarbonate at a temperature sufficient to decompose the sodium bicarbonate to form soda ash as a major product;”

104. *Fu* discloses limitation [5e] for at least the same reasons discussed above regarding claim 3. Section X.D.

vii. [5f] “reacting the dry calcium oxide with the filtered brine effluent to produce ammonia gas and a brine effluent containing calcium chloride wherein said step of reacting the dry calcium oxide with the filtered brine effluent to produce ammonia gas and a brine effluent containing calcium chloride comprises reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone;”

105. *Fu* alone, or *Fu* in view of *Xi* discloses and renders obvious claim limitation [5f] for the same reasons above regarding [1e]. Section X.B.vi.

viii. [5g] “recycling the ammonia gas produced by the reacting step to form additional ammoniated brine using the bubbling ammonia step”

106. *Fu* discloses limitation [5g] for the same reasons above regarding [1f].

Section X.B.vii.

ix. [5h] “crystallizing sodium chloride in the brine effluent; centrifuging the brine effluent to separate the crystallized sodium chloride from the brine effluent;”

107. *Fu* discloses limitation [5h] for the same reasons above regarding [1g].

Section X.B.viii.

x. [5i] “recycling the separated crystallized sodium chloride to form additional fresh brine using the mixing step;”

108. *Fu* discloses or renders obvious limitation 5[i] for the same reasons above regarding [1k]. Section X.B.xii.

xi. [5j] “evaporating water from the effluent to form water vapor and to recover calcium chloride as a major product;”

109. *Fu* discloses limitation [5j] for the same reasons above regarding [1h] and [1i]. Sections X.B.ix-x.

xii. [5k] “collecting the produced calcium chloride;”

110. *Fu* discloses limitation [5k] for the same reasons above regarding [1l].
Section X.B.x.

xiii. [5l] “separating residual salt; and”

111. *Fu* discloses or renders limitation [5l] obvious for the same reasons above regarding [1m]. Section X.B.xiv.

xiv. [5m] “forming a solution wherein the solution is selected from a group consisting of an approximately 77 wt % solution of calcium chloride flakes, and an approximately 95 wt % solution of calcium chloride granules.”

112. *Fu* discloses limitation [5m] for at least the same reasons above regard limitation [1n]. Section X.B.xv. *See also* Section X.G.ii (limitation [6n]).

xv. [5n] “recycling the water vapor to form addition fresh brine using the mixing step;”

113. *Fu* discloses limitation [5n] for the same reasons above regarding [1j] and [1k]. Sections X.B.xi-xii.

xvi. [5o] “whereby all reactants are recycled, leaving substantially no waste to discharge.”

114. *Fu* discloses or renders obvious limitation [5o]. Reactants of the claimed processes are ammonia, sodium chloride, water, calcium oxide, and carbon dioxide. EX1001, claims. As discussed above, *Fu* discloses the recycling of reactants including ammonia (Section X.B.vii), sodium chloride (Sections X.B.viii and X.B.xiv), water (Sections X.B.viii and X.B.xi), and carbon dioxide (Section X.B.iv). In addition, it is my opinion that a POSITA would have understood that calcium oxide is consumed in the soda ash production process and incorporated in the final calcium chloride products, leaving little to recycle. Indeed, nowhere in the '852 Patent specification mentions the recycling of calcium oxide. Because all the remaining reactants are recycled, there is substantially no waste to discharge following the methods disclosed in *Fu*.

115. Alternatively, in my opinion a POSITA would have found it obvious to do so in view of *Fu* and *Xi* because each emphasizes the importance of waste reduction and, *Fu* and *Xi* disclose all of the means of waste reduction described in the '852 Patent.

G. Claim 6

i. [6Pre]-[6m] “A method of producing soda ash and calcium chloride, comprising the steps of: providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine; heating limestone to produce calcium oxide and carbon dioxide; reacting the ammoniated brine with the carbon dioxide produced by the step of heating the limestone to produce sodium bicarbonate, ammonium chloride and a brine effluent; calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide; reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia; recycling the ammonia to be used in the step of ammoniating the brine; and recycling the water from the brine effluent to be used in the step of ammoniating the brine by crystallizing sodium chloride from the mixture of calcium chloride and brine effluent, separating the crystallized sodium chloride from the effluent by centrifuging the effluent, and recycling the crystallized sodium chloride; evaporating any water remaining to produce water vapor; recovering the produced calcium chloride; recycling the water vapor and condensing into water; wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method; collecting the produced calcium chloride; separating residual salt;”

116. *Fu* discloses or renders obvious limitations [6Pre] to [6m] for the same reasons above regarding identical or similar limitations [1Pre] to [1m]. *See* Sections

X.B.i-xiv; Appendix B.

ii. [6n] “and forming an approximately 95 wt % solution of calcium chloride granules.”

117. *Fu* renders obvious limitation [6n]. As discussed above regarding limitation 1[n], *Fu* discloses forming an approximately 77 wt % solution of calcium chloride flakes. Section X.B.xv. Calcium chloride flakes and anhydrous calcium chloride granules, containing approximately 95 wt% of calcium chloride, are two common forms of calcium chloride products. *See, e.g.*, EX1014, p.4 (disclosing both calcium chloride products in “flake form” that contains “approximately 78% calcium chloride” and “anhydrous calcium chloride 94-97%”). In my opinion, it would have been obvious to a POSITA to heat the 78% solution of calcium chloride to further remove the water until an approximately 95 wt% solution of calcium chloride granules (anhydrous calcium chloride) was obtained. A POSITA would understand that anhydrous calcium chloride granules can be used in different applications than those of the calcium chloride flakes and thus would be desirable to produce to broaden the appeal of the calcium chloride products. *See* EX1014 (showing different calcium chloride product offerings). Moreover, it is my opinion that a POSITA would have a reasonable expectation of success because further removal of water from the chloride hydrates produced with *Fu* requires no more than routine practice in the field. Accordingly, *Fu* in view of *Xi* renders claim 6 obvious.

H. Claim 7

i. “The method of producing soda ash and calcium chloride as recited in claim 6, further comprising the step of filtering the mixture of sodium bicarbonate, ammonium chloride, and brine prior to said calcining step, the brine and soluble ammonium chloride being removed for recovery of sodium chloride and ammonia.”

118. Claim 7 depends on claim 6 and recites the same claim limitation as in claim 2. Accordingly, *Fu* renders obvious claim 7 for the same reasons above regarding claims 2 and 6. Sections X.C and X.G.

I. Claim 8

i. “The method of producing soda ash and calcium chloride as recited in claim 7, wherein said step of calcining the sodium bicarbonate comprises heating the filtered sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate.”

119. Claim 8 depends on claim 7 and recites the same claim limitation as in claim 3. Accordingly, *Fu* renders obvious claim 8 for the same reasons above regarding claims 3 and 7. Sections X.D and X.H.

J. Claim 9

i. “The method of producing soda ash and calcium chloride as recited in claim 6, further comprising the steps of: condensing the water vapor; recycling the condensed water vapor, and forming fresh brine with the recycled sodium chloride.”

120. Claim 9 depends on claim 6 and recites the same claim limitation as in claim 4. Accordingly, *Fu* renders obvious claim 9 for the same reasons above regarding claims 4 and 6. Sections X.E and X.G.

K. Claim 10

i. “The method of producing soda ash and calcium chloride as recited in claim 6, wherein said step of reacting the calcium oxide with the ammonium chloride and brine effluent by reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone.”

121. Claim 10 depends on claim 6 and recites a claim limitation similar to [1e]. Accordingly, *Fu* alone, further in view of *Xi*, renders obvious claim 10 for the same reasons above regarding claim 6 and [1e]. Sections X.B.vi and X.G.

XI. GROUND 1B: CLAIMS 6–9 ARE OBVIOUS OVER *FU* IN VIEW OF *HOU*

A. Motivation to Combine

122. As discussed in Sections X.A-B above, *Fu* teaches methods to produce calcium chloride and treat distillation waste liquor and waste residue from the soda ash production process. Similarly, *Hou* also discloses the production of calcium chloride from the waste liquor of soda ash production. EX1006, p.15. It is my opinion that both *Fu* and *Hou* are directed to soda ash production and address similar problem related to the use of waste liquor.

123. *Hou* teaches the benefits of processing calcium chloride hydrates (flakes) to prepare anhydrous CaCl_2 , which “can be used as a catalyst for preparing calcium cyanamide by reacting calcium carbide with nitrogen.” EX1006, p.16. Since calcium chloride flakes and anhydrous calcium chloride granules are two common

forms of calcium chloride products, it is my opinion that a POSITA would have been motivated to combine *Hou*'s further process of calcium chloride hydrates (flakes) with *Fu* to prepare anhydrous CaCl_2 , another desirable form of calcium chloride product. EX1014, p.4 (disclosing both calcium chloride products in "flake form" that contains "approximately 78% calcium chloride" and "anhydrous calcium chloride 94-97%"). In addition, in my opinion, a POSITA would have a reasonable expectation of success because further removal of water from the chloride hydrates produced with *Fu* requires no more than routine practice in the field as taught in *Hou*.

B. Claim 6

i.[6Pre]-[6m]

124. *Fu* disclose or renders obvious limitations [6Pre] to [6m] for the same reasons above regarding identical or similar limitations [1Pre] to [1m]. Sections X.B.i-xiv.

ii.[6n] "and forming an approximately 95 wt % solution of calcium chloride granules."

125. As discussed above, it is my opinion that it would have been obvious for a POSITA to combine *Fu* and *Hou*. Section XI.A. *Fu* discloses a method of producing a 78% solution of calcium chloride. Section X.B.xv; *see also* EX1003, p.79 ("The hot flakes containing the main substance of 78% CaCl_2 are discharged into a cooling rotary cylinder."). *Hou* discloses that "[t]o prepare anhydrous CaCl_2 ,

75% CaCl_2 is heated in a reverberating *Furnace* or a stirring *Furnace*, and the molten substance is frequently raked until part of CaCl_2 is decomposed to generate CaO and HCl . The HCl gas escapes, leaving the molten porous CaCl_2 , called 95% calcium chloride.” EX1006, p.16. Thus, it is my opinion that it would have been obvious to a POSITA to heat the 78% solution of calcium chloride according to *Fu* to obtain the 95% calcium chloride granules using the method disclosed in *Hou*, achieving the benefits of such a product described in *Hou*.

126. Accordingly, *Fu* in view of *Hou* renders obvious claim 6.

C. Claims 7-9

127. *Fu* in view of *Hou* renders obvious claims 7-9. In addition to rendering obvious all limitations of claim 6, *Fu* in view of *Hou* further renders obvious claims 7-9 for the same reasons above discussed in Ground 1A, claims 7-9. Sections X.G-J.

XII. GROUND 1C: CLAIM 10 IS OBVIOUS OVER *FU* IN VIEW OF *Xi* AND *HOU*

128. As discussed above, it is my opinion that a POSITA would have been motivated to combine the teachings of *Fu* with those of *Xi*, or the teachings of *Fu* with those of *Hou* because they all teach the production of calcium chloride from the waste liquor generated during the soda ash production process. In addition, a POSITA would have been similarly motivated to combine *Fu* with *Xi* and further

with *Hou* for the same reasons.

129. *Fu* in view of *Xi* and *Hou* renders obvious claim 10 for the same reasons above discussed in Ground 1A except that *Hou* expressly teaches “forming an approximately 95 wt % solution of calcium chloride granules” as recited in claim 6, which claim 10 depends on. Sections X.G and X.K (claims 6 and 10).

XIII. GROUND 1D: CLAIMS 1–10 ARE OBVIOUS OVER *FU* IN VIEW OF *MOHLER*

A. Motivation to Combine

130. As discussed in Section X.A, *Fu* discloses the method of producing soda ash and calcium chloride and reducing waste “as much as possible” during the process for the sake of environmental protection. *See also* EX1003, p.75-76.

131. *Mohler* is directed to “the recovery of ammonia from aqueous ammonia liquors,” such as the mother liquor that is “obtained in the so-called Solvay or ammonia soda process. EX1007, 1:1-8. *Mohler* also emphasizes that using dry quick lime in the process can reduce water consumption, which also leads to lower energy consumption and less waste. *Id.*, 1:84-106 (emphasis added):

The advantages of the use of dry lime as hereinafter specified include the ***elimination of the step of producing milk of lime*** by the treatment of quick lime with water with a corresponding saving in the operative steps of the process. Again when dry lime is used, since the discharge still liquors are commonly concentrated to recover calcium chloride therefrom, ***a considerable saving in the cost of evaporating the discharge liquors*** results because of the absence of the water contained in the milk of lime. It will also be clear that ***the amount of steam and***

therefore fuel consumption required for operating the still is considerably *decreased* due to the decrease in the volume of liquor treated in consequence of avoiding the water in the milk of lime, and the use of less steam in turn results in less water of condensation being added to the discharge liquors and accordingly further *decreases the cost of concentrating*. There are other advantages attendant upon the use of dry lime, one of which is the *increased capacity of the still* when using dry lime instead of milk of lime.

132. Both *Fu* and *Mohler* are related to soda ash production and address similar problem in connection with waste reduction. In my opinion, a POSITA would have been motivated to combine the soda ash and calcium chloride production process of *Fu* with the teachings of *Mohler* because *Mohler* also teaches the process of recovering ammonia from the mother liquor generated during the ammonia soda process. Further, *Mohler* teaches that using dry quick lime can reduce water consumption and waste generation, which is one of the main goals of *Fu*. See EX1003, p.75-76; EX1007, 1:84-106. It is my opinion that a POSITA would understand that *Mohler's* use of dry quick lime would further the goal of reducing water consumption and waste generation.

133. Therefore, with respect to *Fu's* soda ash and calcium chloride production process, it is my opinion that a POSITA would have been motivated to use dry quick lime as taught by *Mohler* instead of lime milk to react with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. Such a combination represents the use of a known technique (i.e., *Mohler's* use of dry quick lime to recover ammonia from the ammoniated liquor) to

improve a similar process (i.e., *Fu*'s soda ash production process) in the same way. A POSITA would further understand that this modification would not interfere with the other aspects of *Fu*'s processes because the dry lime, upon contact with water in the waste liquor, will effectively turn into lime milk and perform the same functions as intended by *Fu*. Moreover, it is my opinion that a POSITA would have a reasonable expectation of success because replacing lime milk with dry quick lime does not materially change the chemistry involved in Solvay processes and leads to expected benefit of reduced water usage.

B. Claim 1

i.[1Pre]-[1d]

134. *Fu* discloses claim limitations [1Pre]-[1d] for the same reasons above regarding Ground 1A, [1Pre]-[1d]. Section X.B.i-v.

ii. [1e] “reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia; wherein said step of reacting the calcium oxide with the ammonium chloride and brine effluent comprises reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone”

135. *Fu* renders obvious limitation [1e] for the same reasons above regarding Ground 1A. Section X.B.vi.

136. In addition, as described in Section XIII.A, it is my opinion that a POSITA would have been motivated to incorporate *Mohler*'s use of dry quick lime

in *Fu*'s reaction with ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. *Mohler* discloses a process "for using lime in solid form in the recovery of ammonia from solution which comprises packing lime in finely divided form, feeding the compact material through a conduit as a mass of material continuously filling a cross-section of the conduit and advancing the compacted material through an open end of said conduit into the ammonia solution." EX1007, claim 5. *Mohler* also teaches that using solid lime in this process can reduce water consumption and waste generation. See Section XIII.A. It is my opinion that a POSITA would understand the combination of *Fu* and *Mohler* as described above would further their stated goal of reducing water waste since no additional water would be added to slake the lime.

137. Accordingly, it is my opinion that it would have been obvious for a POSITA to combine *Fu* and *Mohler* to directly react the mother liquor with lime powder to produce calcium chloride, brine effluent and ammonia without slaking the calcium oxide in an effort to reduce water consumption in the soda ash production process. *Fu* alone, or *Fu* in view of *Mohler* renders obvious claim 1[e].

iii. [1f]-[1n]

138. *Fu* discloses claim limitations [1f]-[1n] for the same reasons above regarding Ground 1A, [1f]-[1n]. See Sections X.B.vii-xv.

139. Accordingly, *Fu* in view of *Mohler* renders obvious claim 1.

C. Claim 2

140. *Fu* in view of *Mohler* renders obvious claim 2 for the same reasons above regarding claim 1 and claim 2 (Ground 1A). *See* Sections XIII.B and X.C.

D. Claim 3

141. *Fu* in view of *Mohler* renders obvious claim 3 for the same reasons above regarding claim 2 and claim 3 (Ground 1A). *See* Sections XIII.C and X.D.

E. Claim 4

142. *Fu* in view of *Mohler* renders obvious claim 4 for the same reasons above regarding claim 1 and Ground 1A, claim 4. *See* Sections XIII.B and X.E.

F. Claim 5

i. [5Pre]-[5e]

143. *Fu* discloses or renders obvious claim limitations [5Pre]-[5e] for the same reasons above regarding Ground 1A, claim limitations [5Pre]-[5e]. *See* Sections X.F.i-vi.

ii. [5f] “reacting the dry calcium oxide with the filtered brine effluent to produce ammonia gas and a brine effluent containing calcium chloride wherein said step of reacting the dry calcium oxide with the filtered brine effluent to produce ammonia gas and a brine effluent containing calcium chloride comprises reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone;

144. *Fu* alone, or *Fu* in view of *Mohler* renders obvious claim limitation [5f] for the same reasons above regarding [1e]. Section XIII.B.ii.

iii. [5g]-[5o]

145. *Fu* discloses or renders obvious claim limitations [5g]-[5o] for the same reasons above regarding Ground 1A, claim limitations [5g]-[5o]. Sections X.F.viii-xvi.

G. Claims 6-9

146. Claims 6-9 do not recite the limitation regarding the use of dry calcium oxide without slaking. *Fu* in view of *Mohler* discloses or renders obvious claims 6-9 for the same reasons above regarding Ground 1A, claims 6-9. Sections X.G-J.

H. Claim 10

i. “The method of producing soda ash and calcium chloride as recited in claim 6, wherein said step of reacting the calcium oxide with the ammonium chloride and brine effluent by reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone.”

147. Claim 10 depends on claim 6 and recites the claim limitation similar to [1e]. Accordingly, *Fu* alone, further in view of *Mohler*, renders obvious claim 10 for the same reasons above regarding claim 6 and [1e]. Sections X.G. and XIII.B.ii.

XIV. GROUND 1E: CLAIM 10 ARE OBVIOUS OVER *FU* IN VIEW OF *MOHLER* AND *HOU*

148. As discussed above, it is my opinion that a POSITA would have been motivated to combine the teachings of *Fu* with those of *Mohler* to reduce water consumption (Section XIII.A) and to combine the teachings of *Fu* with those of *Hou*

to prepare anhydrous CaCl_2 (Section XI.A). It is my opinion that a POSITA would be further motivated to combine *Fu*, *Mohler*, and *Hou* because they all relate to further processing the waste liquor generated during the soda ash production process.

149. *Fu* in view of *Mohler* and *Hou* renders obvious Claim 10 for the same reasons above discussed in Ground 1C except that *Mohler instead of Xi* expressly teaches “reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone” as recited in claim 10. *See* Sections XIII.G and XII (claims 6 and 10).

XV. GROUND 2: *Du* in View of *Fu* and *Hou* Renders Obvious Claims 1-10

A. Motivation to Combine

150. *Du* discloses a “processing method for preparing calcium chloride solution using filtered mother liquor in ammonia-soda process.” EX1005, Abstract. *Du* identifies the “liquid waste problem” as “crucial” because the waste is “discharged in massive quantity, which is difficult to be handled completely.” *Id.*, [0002]; *see also id.*, [0009].

151. As discussed in Section X.A, *Fu* discloses the method of producing soda ash and calcium chloride and reducing waste “as much as possible” during the process for the sake of environmental protection. *Fu* discloses the recycling of reactants including ammonia (Section X.B.vii), sodium chloride (Sections X.B.viii and X.B.xiv), water (Sections X.B.viii and X.B.xi), and carbon dioxide (Section

X.B.iv).

152. Since *Fu* and *Du* are related to soda ash production and address similar problem in connection with waste reduction, in my opinion, a POSITA would have found it obvious to combine soda ash and calcium chloride production process of *Du* with the teachings of *Fu* to further refine or improve the waste recycling approach.

153. *Du* discloses a separation step of sodium chloride from mother liquor to prepare a saturated ammonium chloride solution. EX1005, [0012]. *Du* further teaches adding dry lime powder directly “into the reactor by a screw conveyor for the reaction with the saturated ammonium chloride solution.” EX1005, [0040].

154. *Fu* discloses reacting calcium hydroxide with ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. In my opinion, it would have been obvious for a POSITA to combine *Du* and *Fu* to react the calcium oxide directly with the ammonium chloride and brine effluent without first removing sodium chloride from the solution. When calcium oxide reacts with ammonium chloride, sodium chloride is not part of the reaction, and it does not impact the reaction in any way, and neither *Du* nor *Fu* teaches otherwise. Therefore, it would have been obvious choices for a POSITA to choose separating sodium chloride either before or after adding CaO, or follow the sequence disclosed in *Fu*.

155. It is my opinion that a POSITA would have a reasonable expectation of

success in combining *Du* and *Fu* and separating the sodium chloride after reacting dry calcium oxide with the mother liquor because the combination merely involves the known chemistry performed in the known way to yield predictable results.

156. Moreover, as discussed above, a POSITA would have been motivated to combine the teachings of *Fu* with those of *Hou* to prepare anhydrous CaCl_2 (Section X.A). A POSITA would be similarly motivated to combine *Du*, *Fu*, and *Hou* because they all relate to further processing the waste liquor generated during the soda ash production process. And such combination would require no more than routine practice in the field.

B. Claim 1

i. [1Pre] “A method of producing soda ash and calcium chloride, comprising the steps of:”

157. *Du* discloses a method of producing soda ash and calcium chloride. *Du* discloses “a method for preparing calcium chloride solution using filtered mother liquor in ammonia soda process, in which the joint production of soda and calcium compound can be achieved....” EX1005, [0011].

158. Accordingly, *Du* discloses 1[pre].

ii. [1a] “providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine;”

159. *Du* discloses limitation [1a]. *Du* discloses providing a source of “sodium chloride solution” (brine) and a source of gaseous “ammonia” in the

“ammonia absorption tower,” where the brine is ammoniated (gaseous ammonia absorbed in the brine) to form ammoniated brine. EX1005, [0024].

iii. [1b] “heating limestone to produce calcium oxide and carbon dioxide;”

160. *Du* alone, or *Du* in view of *Fu*, renders obvious claim limitation [1b]. *Du* discloses that “[d]uring the production of the calcium chloride solution, **lime powder** produced by **grinding in a lime procedure**” is used. EX1005, [0040].

161. *Du* further discloses that other than “distillation for the mother liquor,” “the main procedure for producing soda in ammonia soda process” remains the same as the conventional methods. EX1005, [0011]. It is my opinion that a POSITA would have understood that lime powder produced from “grinding in a lime procedure” refers to dry calcium oxide powder, which, along with carbon dioxide, is routinely obtained from heating limestone. *See also* EX1005, [0017] (“During the production of the calcium chloride solution, lime powder produced by grinding in a lime procedure is pneumatically conveyed into a lime powder storage, and the lime powder is weighed by a material dispenser at the bottom of the storage and sent into the reactor by a screw conveyor for the reaction with the saturated ammonium chloride solution; and the content of active calcium oxide in the lime powder is $\geq 75\%$.”); EX1010, p.8.

162. In addition, *Fu* discloses heating limestone to produce calcium oxide and carbon dioxide. EX1003, p. 52 (“The carbonation process requires a large

amount of CO₂ gas, such that limestone is calcined to produce CO₂ and lime.”). It is my opinion that it would have been obvious for a POSITA to combine *Du* and *Fu* to obtain lime powder and carbon dioxide by heating limestone, according to well-known methods disclosed in *Fu*.

iv.[1c] “reacting the ammoniated brine with the carbon dioxide produced by the step of heating the limestone to produce sodium bicarbonate, ammonium chloride and a brine effluent;”

163. *Du* alone, or *Du* in view of *Fu*, renders obvious limitation [1c]. As discussed in the section above, *Du*, or *Du* in view of *Fu*, renders obvious the claim element “carbon dioxide produced by the step of heating the limestone.”

164. Moreover, *Du* discloses that the ammoniated brine reacts with carbon dioxide to produce sodium bicarbonate, ammonium chloride, and a brine effluent. EX1005, [0025] (“The refined salt solution which has been ammoniated in step 2 is fed to a carbonatization tower, where solid sodium bicarbonate and a filtered mother liquor are formed after the reaction.”), [0008] (“The refined salt solution which has been ammoniated in step 2 is fed to a carbonatization tower, where solid sodium bicarbonate and a filtered mother liquor are formed after the reaction.”).

v.[1d] “calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide;”

165. *Du* discloses limitation [1d]. *Du* teaches that “[t]he separated sodium bicarbonate is calcined into soda.” EX1005, [0027]. In my opinion, it is well-known in the art, and a POSITA would have understood, that when sodium bicarbonate is

calcined, it will decompose and produce soda ash and gaseous carbon dioxide. *See also* EX1013, p.97 (“The reaction in the furnace is the decomposition of sodium bicarbonate into soda ash, carbon dioxide and water”); EX1010, p.8. Alternatively, *Du* in view of *Fu* renders limitation [1d] obvious for the reasons discussed above in Sections X.B.v and XV.A.

vi.[1e] “reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia; wherein said step of reacting the calcium oxide with the ammonium chloride and brine effluent comprises reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone”

166. *Du* alone or further in view of *Fu* renders limitation [1e] obvious. As discussed above regarding [1c], *Du* discloses reacting ammoniated brine with carbon dioxide to produce sodium bicarbonate, ammonium chloride and a brine effluent. *See* Section XV.B.iv.

167. *Du* further discloses producing calcium chloride and ammonia by reacting lime powder with ammonium chloride solution. EX1005, [0031] (“the saturated ammonium chloride solution is sent into a reactor, in which the solution reacts with the condensate produced by ammonia evaporation in the ammonia condenser I and the *lime powder*, and the decomposed reaction solution is fed to a sand separator together with the calcium chloride solution . . . the solution is brought into contact countercurrently with vapor from top to bottom for heat exchange,

thereby *evaporating all the ammonia*”) (emphasis added); *see also id.*, [0017] (“During the production of the calcium chloride solution, lime powder produced by grinding in a lime procedure is pneumatically conveyed into a lime powder storage, and the lime powder is weighed by a material dispenser at the bottom of the storage and sent into the reactor by a screw conveyor for the reaction with the saturated ammonium chloride solution; and the content of active calcium oxide in the lime powder is $\geq 75\%$.”).

168. In my opinion, a POSITA would have understood that lime powder produced by “grinding in a lime procedure” refers to dry calcium oxide powder, because quicklime (lime) is produced through calcination of limestone, which is the baseline for *Du*’s improvement, and *Du* does not mention a slaking process. *See also* EX1003, p.51-52 (“The carbonation process requires a large amount of CO₂ gas, such that limestone is calcined to produce CO₂ and *lime*.”) (emphasis added). Moreover, consistent with the use of dry lime, *Du* teaches that “the lime powder is weighed by a material dispenser at the bottom of the storage and sent into the reactor by a screw conveyor for the reaction.” EX1005, [0017], [0040]. Thus, *Du* discloses “said step of reacting the calcium oxide . . . without slaking the calcium oxide produced by heating the limestone.”

169. To the extent that the Patent Owner argues *Du* does not disclose the brine effluent element because *Du* separates sodium chloride from the mother liquor

before reacting the mother liquor with the lime powder, Patent Owner's argument will not hold. In my opinion, a POSITA would have understood that even though sodium chloride is crystallized and separated from the mother liquor, some residual sodium chloride remains in the mother liquor. EX1001, 2:61-3:7 (disclosing further "separation of salt (NaCl)" after an initial sodium chloride separation step), Claim 1 ("separating the crystallized sodium chloride . . . *separating residual salt.*") (emphasis added). Accordingly, when the ammonium chloride solution reacts with calcium oxide in *Du*, it is my opinion that a POSITA would understand that the ammonium chloride solution also contains brine effluent, which will remain after the reaction.

170. Alternatively, *Du* in view of *Fu* renders limitation [1e] obvious. As discussed above in Section XV.A, *Fu* discloses reacting calcium hydroxide with ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. In my opinion, it would have been obvious for a POSITA to combine *Du* and *Fu* to react the calcium oxide directly with the ammonium chloride and brine effluent without first removing sodium chloride from the solution. When calcium oxide reacts with ammonium chloride, sodium chloride is not part of the reaction, and it does not impact the reaction in any way, and neither *Du* nor *Fu* teaches otherwise. Indeed, *Du* does not disparage or teach away from reacting the calcium oxide with ammonium chloride and brine effluent without first removing sodium

chloride from the solution. Therefore, it is my opinion that it would have been obvious choices for a POSITA to choose separating sodium chloride either before or after adding CaO, or follow the sequence disclosed in *Fu*. A POSITA would have a reasonable expectation of success in combining *Du* and *Fu* and separating the sodium chloride after reacting dry calcium oxide with the mother liquor because the combination merely involves the known chemistry performed in the known way to yield predictable results. *See, e.g.*, EX1008, p.36 (disclosing separating sodium chloride from concentrated calcium chloride based on the known phase diagram of CaCl₂ and NaCl binary system).

vii.[1f] “recycling the ammonia to be used in the step of ammoniating the brine; and”

171. *Du* discloses or renders obvious limitation [1f]. *Du* teaches the disclosed invention “guarantee[s] the recovery and recycling of ammonia and carbon dioxide.” *See* EX1005, Abstract, [0011], [0020]. In my opinion, a POSITA would have understood that the recycled ammonia would be reused in the step of ammoniating the brine, which is key to the ammonia soda process. *See also* Section V.C.

viii.[1g] “recycling the water from the brine effluent to be used in the step of ammoniating the brine by crystallizing sodium chloride from the mixture of calcium chloride and brine effluent, separating the crystallized sodium chloride from the effluent by

centrifuging the effluent, and recycling the crystallized sodium chloride;”

172. *Du* alone or further in view of *Fu* renders limitation [1g] obvious. *Du* teaches crystallizing sodium chloride and separating the crystallized sodium chloride from the effluent. EX1005, [0030] (“The deammoniated mother liquor is fed to a flash tank through a heat exchanger and then is sent into a triple effect evaporator the concentrated mother liquor from the triple effect evaporator is fed to a thickener, and the thickened salt slurry is sent into a separator for separation, thus **obtaining solid sodium chloride** and a filtered salt solution.”) (emphasis added). In my opinion, it is known, and would at least have been obvious, for a POSITA to use a centrifuge as the separator disclosed in *Du* because a centrifuge is routinely used to separate crystallized sodium chloride from effluent by centrifuging the effluent. *See, e.g.*, EX1003, p.77-78; EX1013, p.68.

173. To the extent that *Du* does not disclose “crystallizing sodium chloride from the mixture of calcium chloride and brine effluent,” it would have been obvious for a POSITA to do so as discussed for the claim limitation [1e]. Section XV.B.vi. Because sodium chloride does not participate in the reaction of calcium oxide and ammonium chloride, it is my opinion that a POSITA would have understood that the residual sodium chloride can be separated after the reaction without having any impact on the process. And for the same reasons discussed above in Section XIV.A, it is my opinion that it would have been obvious for a POSITA to modify *Du* in view

of *Fu* to separate crystallized sodium chloride from the calcium chloride and brine effluent after reacting calcium oxide with the mother liquor, thereby recycling sodium chloride.

174. In addition, *Du* teaches “recycling the water from the brine effluent to be used in the step of ammoniating the brine.” For example, *Du* discloses, “During the evaporation and separation of the deammoniated mother liquor, ... the water vapor is directly condensed” (EX1005, [0014]) and “the condensed water is sent into a device for producing soda or calcium compound” (*id.*, [0015]). *See also id.*, [0037], [0038], claims 3 and 4. For the same reasons discussed in Ground 1A above, it would at least be obvious to use the recycled water in the step of ammoniating the brine in order to reduce water consumption of the Solvay process. Section X.B.viii.

ix.[1h] “evaporating any water remaining to produce water vapor;”

175. *Du* discloses or renders obvious limitation [1h]. *Du* discloses that “the joint production of the soda and calcium compound with a concentration of the calcium chloride solution of 30% or more by weight is realized” by using a number of evaporators. EX1005, [0020]; *see also id.*, [0019], [0039]. Alternatively or additionally, it is my opinion that it would have been obvious for a POSITA to evaporate any remaining water to produce water vapor, and to produce the final calcium chloride products as taught by *Fu*. *See also* EX1005, claim 5 (“The processing method for preparing calcium chloride solution using filtered mother

liquor in ammonia-soda process according to claim 1, wherein during the production of the calcium chloride solution, the temperature of the calcium solution after the second flashing is 85°C and the content of calcium chloride in percentage by weight is not less than 30%; fresh vapor with a gauge pressure of 0.25 MPa is used as working vapor and is compressed together with the vapor flashed from the first flash evaporator by means of a vapor injection heat pump and is collected and fed to a bottom ring of the fixed ammonia distillation tower together with vapor having a gauge pressure of 0.07 MPa, where the vapor is guided upwards through a gas separation cap and is brought into contact countercurrently with the reaction flow through tower plates for heat and mass exchange, the evaporated ammonia exits the tower from the top and is collected together with the gas output from the reactor and fed to the condenser I for heat exchange with the filtered mother liquor, the condensed gas is fed to the low vacuum absorption system after cooling through a cooler, and the condensate is sent to the reactor.”); *see also* Section X.B.ix (discussing the term “any water”); Section XV.A.

x.[1i] “recovering the produced calcium chloride;”

176. *Du* discloses limitation [1i]. *Du* teaches that the produced calcium chloride is recovered. *See, e.g.*, EX1005, [0033] (“The calcium solution from the hydrocyclone separator is fed to a clarification tank, in which the solution is settled down by gravity, and a clear top flow flows into a tank for thick calcium solution

and then is pumped by a pump for thick calcium solution to a calcium chloride production device; a bottom flow of thickened slurry is sent by a pressurizing charging pump into a plate-and-frame filter press for pressure filtration, and the filter cake is sent by a belt conveyor to a basic slag dump site, and the qualified filtrate flows into the tank for thick calcium solution.”).

xi.[1j] “recycling the water vapor and condensing into water;”

177. *Du* discloses limitation [1j]. According to *Du*, water vapor is generated in the evaporators and recycled. *See, e.g.*, EX1005, [0038] (“During the evaporation and separation of the deammoniated mother liquor, the condensed water discharged from the heating chamber of the first-effect evaporator and from the reboiler of the free ammonia distillation tower are guided into the flash evaporator, wherein the flashed vapor is fed to the triple effect evaporator and the condensed water is partially sent into an apparatus for temperature maintenance and partially discharged, collected and sent together into a preheater for preheating the mother liquor of raw materials from the second-effect evaporator; the condensed water from the preheater and the condensed water discharged from the heating chamber of the second-effect evaporator are collected in a flash container, the collected condensed water.”), [0039], claims 4 and 5.

xii.[1k] “wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method;”

178. *Du* renders obvious limitation [1k]. As discussed above regarding element [1g], *Du* discloses recovering crystallized sodium chloride, and recycling water by vaporization and condensation. Sections XV.B.viii and XV.B.xi. In my opinion, it would further have been obvious for a POSITA to combine the crystallized sodium chloride with the condensed water to provide fresh brine and continue the method. EX1005, [0009] (“For a person skilled in the art, the second way of treating waste liquid consists in the recycling of the waste liquid back to the sodium carbonate production system itself.”). Alternatively, *Du* in view of *Fu* renders limitation [1k] obvious for the reasons discussed above in Sections X.B.xii and XV.A.

xiii.[1l] “collecting the produced calcium chloride;”

179. *Du* discloses limitation [1l]. *Du* teaches collecting the produced calcium chloride in “the tank for thick calcium solution.” EX1005, [0033].

xiv.[1m] “separating residual salt; and”

180. *Du* alone or further in view of *Fu* renders obvious limitation [1m]. As discussed above, *Du* and *Fu* each discloses separating salt through evaporation and crystallization. Sections XV.B.viii, X.B.viii, X.B.xiv. In my opinion, it would have been obvious for a POSITA to further separate residual salt from the calcium

chloride product to ensure the quality of the calcium chloride produced , at least in view of the express teaching of *Fu*. Section X.B.xiv (disclosing separating residual salt); *see also, e.g.*, EX1008, p.36 (identifying the need of “fully removing salt during calcium chloride making to ensure the quality of both products.”).

xv.[1n] “forming an approximately 77 wt % solution of calcium chloride flakes.”

181. *Du* alone, in view of *Fu*, or further in view of *Hou* renders obvious limitation [1n]. *Du* discloses “achieving a concentration of calcium chloride being 30% (wt) or higher.” EX1005, [0011]. In my opinion, a POSITA would have understood that concentrated calcium chloride in liquid form or in flake form are common commercial types of calcium chloride end products. *See, e.g.*, EX1014, p.4 (disclosing both calcium chloride products in liquid form in concentrations of 28% to 42% and 45%, and in flake form that contains “approximately 78% calcium chloride”). It would have been obvious to further concentrate calcium chloride to arrive at an approximately 77 wt % solution of calcium chloride flakes.

182. Moreover, *Fu* discloses forming calcium chloride flakes with approximately 77 wt% calcium chloride. EX1003, p.79; Section X.B.xv. Similarly, *Hou* discloses “[t]he clarified solution is further concentrated in a cast iron pan until the molten substance solidifies when being cooled to obtain a product called 75% calcium chloride, the corresponding molecular formula of which is $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$.” EX1006, p.15. In my opinion, a POSITA would understand that calcium chloride

flakes containing 75% CaCl_2 is equivalent to approximately 77 wt % solution of calcium chloride flakes. Thus, it would also have been obvious for a POSITA to combine *Du* with *Fu* and/or *Hou* to further increase the concentration of calcium chloride to approximately 77 wt% by evaporating additional water from the calcium chloride solution to achieve the benefits as taught by *Fu* and/or *Hou*.

C. Claim 2

- i. **“The method of producing soda ash and calcium chloride as recited in claim 1, further comprising the step of filtering the mixture of sodium bicarbonate, ammonium chloride, and brine prior to said calcining step, the brine and soluble ammonium chloride being removed for recovery of sodium chloride and ammonia.”**

183. *Du* in view of *Fu* and *Hou* renders obvious claim 2. Further to the discussion above regarding claim 1, *Du* discloses the step of filtering the mixture of sodium bicarbonate, ammonium chloride, and brine prior to the calcining step and the recovery of ammonia from the mother liquor. EX1005, [0026] (“The solid sodium bicarbonate and the filtered mother liquor are separated from each other.”). In addition, *Du* discloses that the brine and soluble ammonium chloride being removed for recovery of sodium chloride and ammonia. *Id.*, [0029] (“the filtered mother liquor from a plant for sodium bicarbonate is heated . . . evaporating all the free ammonia . . . the concentrated mother liquor from the triple effect evaporator is fed to a thickener, and the thickened salt slurry is sent into a separator for separation, thus obtaining solid sodium chloride”).

D. Claim 3

- i. **“The method of producing soda ash and calcium chloride as recited in claim 2, wherein said step of calcining the sodium bicarbonate comprises heating the filtered sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate.”**

184. *Du* in view of *Fu* and *Hou* renders obvious claim 3. Further to the discussion above regarding claims 1 and 2, *Du* teaches that “[t]he separated sodium bicarbonate is calcined into soda [ash].” EX1005, [0027]. Calcination of sodium bicarbonate is known to involve heating the sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate. *Fu* expressly teaches that “[s]odium bicarbonate is an unstable compound, and can be decomposed at a room temperature and rapidly decomposed by increasing the temperature. ... when the temperature is 100-101°C, the decomposition pressure has reached 0.1 MPa, that is, NaHCO₃ can be completely decomposed. ...in fact, the soda ash discharging temperature of the calcination is mostly controlled at 165-190°C.” EX1003, p.68. Alternatively, it is my opinion that it would have been obvious to heat the filtered sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate as taught by *Fu*. *See id.*

E. Claim 4

- i. **“The method of producing soda ash and calcium chloride as recited in claim 1, further comprising the steps of condensing the**

water vapor and recycling the condensed water vapor for the formation of fresh brine with the recycled sodium chloride.”

185. As discussed above regarding limitation [1k], *Du* renders it obvious that the recycled sodium chloride is used for the providing of fresh brine by combining with the condensed water vapor. At least for the same reasons discussed in Section XV.B.xii, *Du* in view of *Fu* and *Hou* renders obvious claim 4.

F. Claim 5

i. [5Pre] “A method of producing soda ash and calcium chloride, comprising the steps of:”

186. *Du* disclosed a method of producing soda ash and calcium chloride for the same reasons above regarding [1Pre]. *See* Section XV.B.i.

ii. [5a] “mixing sodium chloride and water to form fresh brine; providing ammonia gas; bubbling the ammonia gas through the fresh brine to form ammoniated brine;”

187. *Du* discloses or renders obvious limitation [5a]. *Du* discloses that “the ammonia must be absorbed [] with the sodium chloride solution” in an “ammonia absorption tower.” EX1005, [0024]. In my opinion, a POSITA would have understood that fresh brine can be obtained by mixing sodium chloride and water and ammonia is absorbed in the ammonia absorption tower by bubbling gas through the fresh brine, which is a routine practice in the industry. *See, e.g.*, EX1013, p.13 (showing an ammonia gas absorption tower), p.63 (“These center holes were also provided with teeth to subdivide and distribute the gas bubbles in the column.”)

188. Additionally, the combination of *Du* and *Fu* further discloses this limitation. As discussed in Section X.F.ii, *Fu* discloses bubbling the ammonia gas through the fresh brine to form ammoniated brine.

iii. [5b] “heating limestone to produce dry calcium oxide and carbon dioxide;”

189. *Du* alone, or *Du* in view of *Fu*, renders obvious limitation [5b] for the same reasons above regarding [1b]. Section XV.B.iii.

iv. [5c] “bubbling the carbon dioxide through the ammoniated brine in a carbonation tower to produce sodium bicarbonate, ammonium chloride, and a brine effluent, the sodium bicarbonate precipitating in the brine effluent and the ammonium chloride being soluble in the brine effluent;”

190. *Du* discloses or renders obvious limitation [5c]. *Du* discloses that “in the ammonia absorption tower, the ammonia must be absorbed first with the sodium chloride solution before the absorption of carbon dioxide” and “[t]he refined salt solution which has been ammoniated in [the ammoniating step] is fed to a carbonatization tower, where solid sodium bicarbonate and a filtered mother liquor are formed after the reaction.” EX1005, [0024], [0025]. In my opinion, a POSITA would have understood that the carbon dioxide is bubbled through the ammoniated brine in the carbonation tower, which is a routine practice in the industry. *See, e.g.*, EX1013, p.34 (showing a CO₂ gas absorption tower).

191. *Du* further discloses “the sodium bicarbonate precipitating in the brine

effluent and the ammonium chloride being soluble in the brine effluent.” EX1005, [0026] (“The solid sodium bicarbonate and the filtered mother liquor are separated from each other.”), [0008] (“The filtered mother liquor contains mainly ammonium chloride as well as unconverted sodium chloride and ammonium bicarbonate”).

v. [5d] “filtering the brine effluent to remove the precipitated sodium bicarbonate”

192. *Du* discloses limitation [5d]. *Du* discloses that “[t]he solid sodium bicarbonate and the filtered mother liquor are separated from each other.” EX1005, [0026].

vi. [5e] “calcining the filtered sodium bicarbonate at a temperature sufficient to decompose the sodium bicarbonate to form soda ash as a major product;”

193. *Du* discloses limitation [5e] for at least the same reasons discussed above regarding claim 3. Section XV.D.

vii. [5f] “reacting the dry calcium oxide with the filtered brine effluent to produce ammonia gas and a brine effluent containing calcium chloride wherein said step of reacting the dry calcium oxide with the filtered brine effluent to produce ammonia gas and a brine effluent containing calcium chloride comprises reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone;”

194. *Du* alone, or *Du* in view of *Fu* renders obvious claim limitation [5f] for the same reasons above regarding [1e]. Section XV.B.vi.

- viii. **[5g] “recycling the ammonia gas produced by the reacting step to form additional ammoniated brine using the bubbling ammonia step”**

195. *Du* discloses or renders obvious limitation [5g] for the same reasons above regarding [1f]. Section XV.B.vii.

- ix. **[5h] “crystallizing sodium chloride in the brine effluent; centrifuging the brine effluent to separate the crystallized sodium chloride from the brine effluent;”**

196. *Du* discloses or renders obvious limitation [5h] for the same reasons above regarding [1g]. Section XV.B.viii.

- x. **[5i] “recycling the separated crystallized sodium chloride to form additional fresh brine using the mixing step;”**

197. *Du* renders obvious limitation [5i] for the same reasons above regarding [1k]. Section XV.B.xii.

- xi. **[5j] “evaporating water from the effluent to form water vapor and to recover calcium chloride as a major product;”**

198. *Du* discloses limitation [5j] for the same reasons above regarding [1h] and [1i]. Sections XV.B.ix-x.

- xii. **[5k] “collecting the produced calcium chloride;”**

199. *Du* discloses limitation [5k] for the same reasons above regarding [1l]. Section XV.B.x.

- xiii. **[5l] “separating residual salt; and”**

200. *Du* discloses or renders limitation [5l] obvious for the same reasons above regarding [1m]. Section XV.B.xiv.

- xiv. [5m] **“forming a solution wherein the solution is selected from a group consisting of an approximately 77 wt % solution of calcium chloride flakes, and an approximately 95 wt % solution of calcium chloride granules.”**

201. *Du* in view of *Fu* and *Hou* renders obvious limitation [5m] for at least the same reasons above regard limitation [1n]. Section XV.B.xv; *see also infra* Section XV.G (limitation [6n]).

- xv. [5n] **“recycling the water vapor to form addition fresh brine using the mixing step;”**

202. *Du* renders obvious limitation [5n] for the same reasons above regarding [1j] and [1k]. Sections XV.B.xi-xii.

- xvi. [5o] **“whereby all reactants are recycled, leaving substantially no waste to discharge.”**

203. *Du* in view of *Fu* renders obvious limitation [5o]. Reactants of the claimed processes are ammonia, sodium chloride, water, calcium oxide, and carbon dioxide. EX1001, claims. As discussed above, *Fu* and *Du* each discloses the recycling of reactants including ammonia, sodium chloride, water, and carbon dioxide. *Du* also “guarantee[s] the recovery and recycling of ammonia and carbon dioxide.” EX1005, Abstract. In addition, it is my opinion that a POSITA would have understood that calcium oxide is consumed in the soda ash production process and incorporated in the final calcium chloride products, leaving little to recycle. Because all the remaining reactants are recycled, there is substantially no waste to discharge following the methods disclosed in *Fu* and/or *Du*. *See also* EX1005, [0009] (“For a

person skilled in the art, the second way of treating waste liquid consists in the recycling of the waste liquid back to the sodium carbonate production system itself.”).

204. Alternatively, it is my opinion that a POSITA would have found it obvious to do so in view of *Du* and *Fu* because each emphasizes the importance of waste reduction and, *Du* and *Fu* disclose all of the means of waste reduction described in the '852 Patent.

G. Claim 6

i. [6Pre]-[6m] “A method of producing soda ash and calcium chloride, comprising the steps of: providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine; heating limestone to produce calcium oxide and carbon dioxide; reacting the ammoniated brine with the carbon dioxide produced by the step of heating the limestone to produce sodium bicarbonate, ammonium chloride and a brine effluent; calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide; reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia; recycling the ammonia to be used in the step of ammoniating the brine; and recycling the water from the brine effluent to be used in the step of ammoniating the brine by crystallizing sodium chloride from the mixture of calcium chloride and brine effluent, separating the crystallized sodium chloride from the effluent by centrifuging the effluent, and recycling the crystallized sodium chloride; evaporating any water remaining to produce water vapor; recovering the produced calcium chloride; recycling the water vapor and condensing into water; wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method;

collecting the produced calcium chloride; separating residual salt;”

205. *Du* alone, or *Du* in view of *Fu*, renders obvious limitations [6Pre] to [6m] for the same reasons above regarding identical or similar limitations [1Pre] to [1m]. *See* Sections XV.B.i-xiv; Appendix B.

ii. [6n] “and forming an approximately 95 wt % solution of calcium chloride granules.”

206. *Du* discloses “achieving a concentration of calcium chloride being 30% (wt) or higher.” EX1005, [0011]. In my opinion, a POSITA would have understood that concentrated calcium chloride in liquid form or in anhydrous form are common types of calcium chloride end products. *See, e.g.*, EX1014, p.4 (disclosing both calcium chloride products in liquid form in concentrations of 28% to 42% and 45%, and in “anhydrous calcium chloride 94-97% mini-pellets”). It would have been obvious to further concentrate calcium chloride to arrive at an approximately 95 wt % solution of calcium chloride granules.

207. In addition, *Hou* discloses that “[t]o prepare anhydrous CaCl_2 , 75% CaCl_2 is heated in a reverberating *Furnace* or a stirring *Furnace*, and the molten substance is frequently raked until part of CaCl_2 is decomposed to generate CaO and HCl . The HCl gas escapes, leaving the molten porous CaCl_2 , called 95% calcium chloride.” EX1006, p.16. In my opinion, it would also have been obvious for a POSITA to combine *Du* with *Fu* and *Hou* to further increase the concentration of

calcium chloride to approximately 95 wt% by evaporating additional water from the calcium chloride solution, achieving the benefits of such a product described in *Hou*.

208. Accordingly, *Du* in view of *Fu* and *Hou* renders obvious claim 6.

H. Claim 7

- i. “The method of producing soda ash and calcium chloride as recited in claim 6, further comprising the step of filtering the mixture of sodium bicarbonate, ammonium chloride, and brine prior to said calcining step, the brine and soluble ammonium chloride being removed for recovery of sodium chloride and ammonia.”**

209. Claim 7 depends on claim 6 and recites the same claim limitation as in claim 2. Accordingly, *Du* in view of *Fu* and *Hou* renders obvious claim 7 for the same reasons above regarding claims 2 and 6. *See* Sections XV.C and XV.G.

I. Claim 8

- i. “The method of producing soda ash and calcium chloride as recited in claim 7, wherein said step of calcining the sodium bicarbonate comprises heating the filtered sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate.”**

210. Claim 8 depends on claim 7 and recites the same claim limitation as in claim 3. Accordingly, *Du* in view of *Fu* and *Hou* renders obvious claim 8 for the same reasons above regarding claims 3 and 7. *See* Sections XV.D and XV.H.

J. Claim 9

- i. “The method of producing soda ash and calcium chloride as recited in claim 6, further comprising the steps of: condensing the**

water vapor; recycling the condensed water vapor, and forming fresh brine with the recycled sodium chloride.”

211. Claim 9 depends on claim 6 and recites the same claim limitation as in claim 4. Accordingly, *Du* in view of *Fu* and *Hou* renders obvious claim 9 for the same reasons above regarding claims 4 and 6. *See* Sections XV.E and XV.G.

K. Claim 10

i. “The method of producing soda ash and calcium chloride as recited in claim 6, wherein said step of reacting the calcium oxide with the ammonium chloride and brine effluent by reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone.”

212. Claim 10 depends on claim 6 and recites the claim limitation similar to [1e]. Accordingly, *Du* in view of *Fu* and *Hou* renders obvious claim 10 for the same reasons above regarding claim 6 and [1e]. *See* Sections XV.G and XV.B.vi.

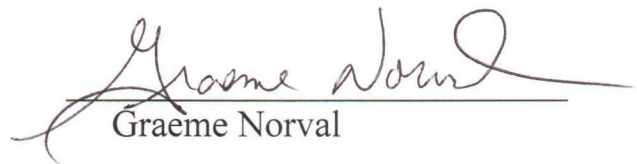
XIV. Reservation of Rights

My opinions are based upon the information that I have considered to date. I am unaware of any evidence of objective indicia with respect to the '852 Patent that would render any of the challenged claims non-obvious. I reserve the right, however, to supplement my opinions in the future to respond to any arguments raised by the owner of the '852 Patent and to consider new information that becomes available to me.

XVI. DECLARATION

213. All statements made herein of my own knowledge are true, and all statements made on information and belief are believed to be true. I further understand that willful false statements and the like are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code. I declare under penalty of perjury that the foregoing is true and correct.

Executed on December 16, 2022.



Graeme Norval

Appendix A

Graeme W. Norval, Ph.D., F.C.I.C., P.Eng.

Curriculum Vitae

Address

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Key Expertise Areas

My expertise is with Inorganic Process Chemistry and Engineering, and in particular, the commodity chemicals - chlorine, caustic soda, hydrochloric acid, sodium chlorate, sodium hypochlorite, sulfuric acid, and liquid sulfur dioxide. I have worked on projects investigating the aqueous solution chemistry as well as manufacture, distribution and usage of these products. Examples include the preparation of pure NaCl brines, and their electrolytic conversion into chlorine and caustic soda, and the process engineering required to convert these into market-grade products. I have performed troubleshooting projects for liquid chlorine pipelines, as well as developing carbo-chlorination technology. I have developed processes for treating the weak sulfuric acid effluent generated at base metals smelters, as well as developing technology for the solution phase treatment of industrial spent sulfuric acids. Through working with these varied chemicals and industries, I have gained a strong understanding of Process Safety management.

Employment History

2016 – 2021 Professor, Teaching Stream; Department of Chemical Engineering and Applied Chemistry, University of Toronto
2010 - 2016 Senior Lecturer and Associate Chair and Undergraduate Coordinator, Department of Chemical Engineering and Applied Chemistry, University of Toronto
2007 - 2010 Lecturer and Associate Chair and Undergraduate Coordinator, Department of Chemical Engineering and Applied Chemistry, University of Toronto
2002 - 2007 Lecturer, Department of Chemical Engineering and Applied Chemistry, University of Toronto

2002 - Principal of GWN Chemical Consulting, Inc.
1995 – 2002 Senior Research Engineer, PCI Chemicals Canada Inc., Pioneer Technology Centre
1989 – 1995 Research Engineer, ICI Forest Products (predecessor of PCI Chemicals Canada Inc.)

Professional Activities

I am a Licensed Professional Engineer with Professional Engineers Ontario (PEO).

I am the Secretary to the Board of Directors of the Canadian Catalysis Foundation, a non-profit organization supporting catalysis research in Canada.

Education

Ph.D. in Chemical Engineering, University of Toronto, 1989, Reaction Engineering

M.A.Sc. in Chemical Engineering, University of Toronto, 1985, Heterogeneous Catalysis

B.A.Sc. in Chemical Engineering, University of Toronto, 1983

Awards

2017 – Faculty of Applied Science and Engineering, Sustained Teaching Excellence Award

2013 - Process Safety Management Award winner, Canadian Society for Chemical Engineering

1993 - ICEE (Innovation, Creativity and Excellence in Engineering), ICI Engineering

Hobbies

I am an avid golfer at Rattlesnake Point Golf Club, and an organic gardener.

Safety Management Related Activities

I was a member of the Prevention Council, an advisory board to the Ontario Minister of Labour between 2012 and 2020, including being Chair between 2015 and 2020. This Prevention Council was created as part of a focus shift from enforcement of safety infractions, to one of prevention of injuries and accidents. Key outcomes were the development of mandatory awareness training for all workers and supervisors, and a shift to the use of data and evidence for regulation development.

I chaired the CSA Z-767 Process Safety Management Technical Committee between 2015 and 2021. During that time, the Z-767 Process Safety Management standard was developed and published. I continue with the Technical Committee as Vice-Chair, as we work on revisions to the standard.

I am an active member of the Process Safety Management Division of the Canadian Society for Chemical Engineering.

Key Technical Achievements

I have assisted in the design of a dechlorination system for the Metropolitan Water District of Southern California. Metropolitan operates the only chlorine trailer fleet in North America, and the trailers are transloaded from rail cars at a specially built facility. Vent gases are sent through a water aspirator to the Colorado River Aqueduct, and the residual chlorine must be destroyed to meet Fish and Game regulations in the downstream lakes.

I identified the root cause of high CO₂ in pharmaceutical grade NO gas, and worked with the client's quality management team to implement preventive purchasing arrangements, as well as an equipment upgrade. NO gas is used to treat “blue babies”, and improve their lung capacity.

I have prepared the Emission Summary and Dispersion Models and application packages for Certificates of Approval (Air) for 12 clients in southern Ontario. I have completed registration packages for PMRA (Pesticide Management Regulatory Agency) registrations for sanitizers and horticultural vinegar products.

I acted as expert witness for a valve supplier who was being sued by a major chemical producer. The issue revolved around fitness for service. The valves were a modified (smaller) version of a standard European rail car angle valve. The valve had a spring-loaded ball check valve installed underneath, such that in the event of a roll-over with loss of the valve, there is no leak. The valves caused cavitation because the chemical producer was filling at a flowrate that was 40% greater than the design value for the valves. The client was found to have no responsibility in a public trial.

I worked with a polymer manufacturer on the safe injection of organic sulfates which improved the properties of the PET resin.

I worked as part of the joint technical team tasked with resolving a corrosion issue within a 7-mile chlorine pipeline. A mathematical model of the system was developed, which predicted the variation in pressure drop with operating rates and time on stream. A series of solutions were implemented at no new capital outlay, and there have been no problems since implementation.

A sodium hypochlorite repackager experienced several episodes of swelling bleach bottles leading to bottles falling off the shelves in the stores. I directed a study that demonstrated that the sodium hypochlorite was not decomposing into O₂. The root cause was a flaw in the bleach bottle design, and the use of a gas impermeable seal. The customer has since modified their bottle sealing operation, and the problem has never returned.

I was part of a 3-person team that won the 1993 ICEE (Innovation, Creativity and Excellence in Engineering), awarded by ICI Engineering. The team identified a low cost method for reducing the dissolved gas content in liquid chlorine. The method was implemented for one of our customers, and enabled ICI to retain the contract with a high net-back customer, at no capital expense.

Summary of Recent Consulting Engineering Experience

This table provides information on a number of the projects on which I have worked over the past 10 years.

Location	Name of Client & Contact Person	Completion Date	Description of my Duties
Bécancour, Québec	Olin Canada, Hélène Bédard	Ongoing since 2002	Client manufactures chlorine and caustic soda; I provide consulting services on chlorine operation; bleach chemistry and operation; caustic soda evaporation; brine treatment
Oakville, Ontario	Kencro Chemicals (division of UBA); Denis Manias	Ongoing since 2002	Client repackages inorganic chemicals; I provide technical information and advice on product and packaging issues; completed the Environmental Compliance Approval (multiple revisions over the years)
St Michel-des Saints, Québec	Nouveau Monde Graphite; Alain Mercier	Ongoing since 2021	Client is working on mining graphite, followed by purification with chlorine, in order to supply the lithium battery market. I am the chlorine expert, supporting the development of the process design.
Dundas, Ontario	Darling International; Abraham Mun	Ongoing since 2020	Client operates multiple rendering facilities in Canada. I provide support and expert training on the chemistry involved in the odour abatement scrubbers as well as the effluent treatment system
London, Ontario	P.J. Holdings, Inc.; Phil Pecoskie	Ongoing since 2015	Client is a producer of hypochlorous acid, a pH neutral hypochlorite solution for disinfection. I have managed the application for a DIN registration, including working with the microbiologists doing the laboratory studies as well as developed the supporting application documents. I also have provided technical and training support for the commercialization of the products

New York, New York	Jos. H. Lowenstein & Sons; Dr. Chao Zheng	December 2018	Client receives resorcinol from a new supplier; it is contaminated with a thiol; I am working on odour abatement at the facility, as well as treatment at the manufacturer's site
Mississauga, Ontario	Hathaway Research International; George Hathaway	August 2018	Client is interested in a process to manufacture polysilazanes; a process flowsheet was developed, along with basic equipment sizing; client was connected with a pilot plant manufacturer for detailed design and fabrication
Thetford Mines, Québec	Les consultants t.a.g. inc; Yvan Huard	November 2018	Client is interested in manufacturing muriatic acid for the production of solutions of inorganic chlorides
Toronto, Ontario	ERCO; John Bishop	April 2018	A model of the chlorine compression system was created so as to determine whether NCl ₃ could accumulate in a liquid phase (NCl ₃ is detonable)
Pittsburg, Pennsylvania	Marcus & Shapira, LLP; Joshua Kobrin	February 2018	I provided expert witness services (analyzed information, wrote report, testify in trial) in a case involving angle valves for chlorine rail cars
Toronto, Ontario	Aquabond, Bruce Girard	February 2017	I developed the application package for an environmental compliance approval; managed it through to approval
Pittsburg, Pennsylvania	Fox Rothschild LLP; Patrick Abranowich	July 2016	Provided expert witness services (developed report) for a case involving a change in chlorine supply to a TiO ₂ producer which adversely impacted one of their clients disproportionately
Salinas, California	Dole Foods, Terry Young	February 2016	Client packages various salad products; I worked to improve food safety provided technical services on water analysis, operation of water softening, pH and ORP analyses;
Bécancour, Québec	Air Liquide, Richard Pawulski	November 2015	I worked with client team to develop a process to remove chloramines from hydrogen gas; equipment started up in early 2015.
Newark, New Jersey	Sieman's, James Kegelman	July 2015	Client markets analytical instruments, and supplies small packages of dilute

			sodium hypochlorite; from time to time - the bottles swell; I provided consulting services on the root cause of the swelling, as well as how to prevent the issue; worked with client on changes to the bottles
Bloomfield, Ontario	Universal Fan & Blower; Arnold Robb	August 2014	Client produces corrosion resistant fans for chemical environments; I developed the application for an Environmental Compliance Approval and managed the process through to approval
London, UK	Stephenson Harwood LLP; Cristan Evans	May 2014	Acted as expert witness (analyzed information, write report, testified) in a case settled under Maritime Law; the case involved the definition of “industrial grade” sulfuric acid, and the normally expected impurities
Sarnia, Vaughan, Ontario	Ershigs; Caroline Peters	2010 – 2014	The client manufactures vessels, pipe and fittings from fibreglass reinforced polymers (FRP). I developed and submitted the application packages for Environment Compliance Approvals for the 2 facilities.

List of Publications

Pawulski, R., S. Houle, B. Fillion, B., Y. Chen, G. Norval, O. Napotchatyckh., “Purification of Hydrogen”, submission to US Patent Office

Norval, G.W., “System and Method for the Purification and Recovery of Potash”, US Patent Submission

Stuart, A.T.B., A.I. Miller and G.W. Norval, “Distributed Pre-enrichment Method and Apparatus for Production of Heavy Water”, WO2011011876A1, February 3, 2011.

Stuart, A.T.B., A.I. Miller and G.W. Norval, “Chlorate and Chlorine Dioxide Systems Adapted for the Production of Deuterium Enriched Water”, CA 2769142, February 3, 2011.

Dijkstra, R., D. Lawler, C.M. Evans, and G.W. Norval, “Process for Purifying Waste Sulfuric Acid”, CA2646311A1, June 11, 2010.

Norval, G.W. and D.J. Drexler, “NO_x Removal from Sulfur Dioxide Containing Gases”, Canadian Patent 2,277, 678 (2008).

Sturgeoff, L.G., G.W. Norval, F.S. Przystal and A.E. Yethon, “Fluoride Removal from Sulfuric Acid”, U.S. Patent 5,324,499 (1994).

The first patent is the result of consulting work. Byproduct hydrogen from a chlorine plant can be contaminated with chloramines. These are an issue if the hydrogen is to be used in hydrogen peroxide manufacture (they react with the solvent). The patent discloses the use of a fixed bed activated carbon treatment system to remove the chloramines prior to hydrogen processing.

The second patent is the result of work with a group trying to build sustainable agriculture in Africa. Cocoa is rich in potassium; the cocoa pod husk is 60% of the dry mass of the cocoa fruit, and is an agricultural waste. The husk is discarded, as it leads to a viral infection of the trees. We are working to convert this biomass to energy, which destroys the virus as well as provides local electricity. Further, we produce potassium carbonate from the ash, which can be used as fertilizer, or potassium alkali.

The next pair of patents results from consulting work. The manufacture of heavy water is expensive - both capital and operating costs. These patents work to optimize the cost factors, using a centralized heavy water distillation plant, with remote producers being hydrogen plants of any kind. More specifically, a sodium chlorate (or integrated ClO₂ facility) is a water electrolysis facility, which is well suited to minor modifications to permit segregation of a D₂O elevated water stream.

The Dijkstra et. al. patent deals with recovering the waste sulfuric acid generated in organic silicones manufacture, which contains a substantial amount (~20 wt%) of dissolved dimethyl ether. The process allows for the recovery of the ether, followed by recovery of a clean concentrated sulfuric acid stream. This technology has been installed in 2 facilities in Asia and a North American producer is working to implement the technology.

The NO_x removal patent deals with NO_x elimination in a metallurgical sulfuric acid plant. NO_x dissolved in sulfuric acid gives nitrosylsulfuric acid, which is an unwanted contaminant for numerous reasons. Gas phase destruction avoids the problem and cost of injecting expensive reducing agents into liquid sulfuric acid. This technology has been installed in a Canadian and a European metallurgical sulfuric acid plant.

The final patent deals with removal of HF from sulfuric acid, and relies on managing concentration and temperature to allow stripping the HF. The technology has been installed in a European metallurgical sulfuric acid plant.

Refereed Publications

Norval, G.W. and A.W. Chan, “Gender Differentials on Academic Performance and Lifelong Learning Attribute in Chemical Engineering”, *J. Chem. Educ.* 99(1) 252-258 (2022).

Kone, K., Akueson, K., and G. Norval “On the Production of Potassium Carbonate from Cocoa Pod Husks”, *Recycling* 5(3), 23-28 (2020).

Norval, G.W., “Chronic Lung Impact on Laboratory Worker Exposed to Chloramines and Cyanogen Chloride”; *ACS Chem. Health Saf.* 27 (2), 129-132 (2020).

Norval, G.W., “Analysis of a Blast due to Inadvertent Mixing of Ammonium Sulfate and Sodium Hypochlorite”, *Proc. Safety Prog.* 35(1) 92-95 (2016).

Norval, G.W., “Aspects Of Changing The Safety Culture In Today's Universities”, *Can. J. Chem. Eng.* 93(3) 1154–1159 (2015).

Maka, K., and G.W. Norval, “The Optimum Design of a Chlorine Compression and Liquefaction System, Including NCl_3 Destruction”, *Can. J. Chem. Eng.* 91(4) 718-724 (2013).

Norval, G.W., “Notes on the Issues of Equilibrium in the Fischer-Tropsch Synthesis”, *Can. J. Chem. Eng.* 86 1062-1069 (2008).

R. Dufresne, L. Caouette, G.W. Norval and C.J. Kanters, “ Treatment of Clean Condensate using Catalytically Enhanced Oxidation”, *Pulp Paper Can.* 102 T39-T42 (2001).

Norval, G.W., M.J. Phillips, R.W. Missen and W.R. Smith, “Identification and Application of Partial Equilibrium in Reactor Modelling”, *A.I.Ch.E.* 38 1288-1293 (1992).

Norval, G.W., M.J. Phillips, R.W. Missen and W.R. Smith, “Constrained Chemical Equilibrium and Incompletely Specified Elemental Abundance Data”, *Can. J. Chem. Eng.* 69 1184-1192 (1991).

Norval, G.W., and M.J. Phillips, “Application of Equilibrium Analysis to a Fischer-Tropsch Product”, *J. Catal.* 126 87-91 (1990).

Norval, G.W., M.J. Phillips, R.W. Missen and W.R. Smith, “Calculated Equilibrium for the Alkene and Alcohol Aromatization Processes”, *Appl. Catal.* 54 37-52 (1989).

Norval, G.W., M.J. Phillips, R.W. Missen and W.R. Smith, “On Chemical Equilibrium for the Pseudo-One-Element Systems”, *Can. J. Chem. Eng.* 67 652-657 (1989).

Norval, G.W., M.J. Phillips, R.W. Missen and W.R. Smith, "Application of Equilibrium Sensitivity Analysis to Aromatization Processes", I. & E.C. Research 28 1884-1887 (1989).

Norval, G.W., and M.J. Phillips, "Mossbauer Spectroscopy Study of Iron Ammonia Synthesis Catalysts", J. Phys. Chem. 90 4743-4747 (1986).

Refereed Research Notes

Norval, G.W., M.J. Phillips, K.S. Virk and R.V. Simons, "Olefin Conversion over Zeolite ZSM-5", Can. J. Chem. Eng. 67 521-523 (1989).

Norval, G.W., and M.J. Phillips, "Effect of Promoters on Benzene Hydrogenation over Fused Iron Catalysts", J. Catal. 115 250-253 (1989).

Norval, G.W., C. Crimi, and M.J. Phillips, "The Conversion of Propene and Ethene over AlPO₄-11", J. Catal. 113 263-265 (1988).

Norval, G.W., and M.J. Phillips, "Alkylation of Toluene with Ethylene", React. Kinet. Catal. Lett. 38 261-264 (1986).

Letter to the Editor

Norval, G.W. and M.J. Phillips, "Comment on Induction Periods for Synthesis of Hydrocarbons from Syngas over Metal/Zeolite Catalysts Using a Two-Stage Process", Appl. Catal. 43 193-195 (1988).

Unrefereed Publications

Norval, G.W., T. Burton and C. Kanters, "The Removal of Pulp Mill Odours by Novel Catalytic Environmental Technology", Pulp Paper Can. 102 T111-T113 (2001).

C.M. Evans, E.G. Lyne and G.W. Norval, "Treatment of Weak Acid Effluent from Metallurgical Acid Plant Gas Cleaning Systems", in Materials Processing Technology and Environmental Protection in Mining and Metallurgy, Godbehere, P.H., V. Samuel, M. Iwanec, and P. Barr (Eds), The Metallurgical Society of CIM, Montreal, Quebec, 1993, p107-123.

Norval, G.W. and B.A. Edwards, "Pilot Plant Aids Pulp Industry", The ICI Engineer 15 6 (1992).

Conference Presentations

Norval, G.W., “CAN/CSA 767-17”, Compressed Gas Association AGM, Ottawa, September 10, 2019.

Norval, G.W., “Teaching Safety in Engineering Education”, Conseil pour la Reducation des Accidents Industriels Majeurs AGM, Montréal, January 30, 2020 (in French).

Norval, G.W., Bobicki, E., Continuous Improvements In Third Year Chemical Engineering Design, CEEA Ottawa, June 11, 2019.

Castrucci, J., Jadidian, P, Norval, G.W., “E-Learning for Safety Education in Engineering”, CSCHE 69th Meeting, Halifax, October 21, 2019.

Norval, G.W., Szabo, P., Jowlabar, P. and G. Wilson “A New Approach to the Unit Operations Laboratory at the University of Toronto”, 2nd Canadian Engineering Education Association Conference, St. John’s Newfoundland, June 2011.

Norval, G.W., Pasteris, A.E., and V. Pakalnis “Teaching of Health and Safety in the Undergraduate Engineering Curriculum”, 1st Canadian Engineering Education Association Conference, Kingston, Ontario, June 2010.

Norval, G.W., “Improving the Teaching of Safety Health and the Environment”, Process Safety and Loss Management Symposium, 58th Canadian Society for Chemical Engineering Conference, Ottawa, Ontario, October 2008.

Norval, G.W., “A New Course in Chemical Engineering Design”, 55th Canadian Chemical Engineering Conference, Toronto, Ont. 2005.

Norval, G.W., S.W. Park and J.P. Gaudreault, “Testing of Materials for use in a Cl₂-Saturated CHCl₃ System”, 51st Canadian Chemical Engineering Conference, Halifax, October 2001.

Norval, G.W., J.T. Burton and C.J. Kanters, “The Removal of Pulp Mill Odours by Novel Catalytic Environmental Technology”, 86th Annual Meeting of the Pulp and Paper Association of Canada, Montreal, QC, February 2000.

R. Dufresne, L. Caouette, G.W. Norval and C.J. Kanters, “Treatment of Clean Condensate using Catalytically Enhance Oxidation”, 86th Annual Meeting of the Pulp and Paper Association of Canada, Montreal, QC, February 2000, and at the 16th Canadian Symposium on Catalysis, Banff, Alberta, May 2000 and Conférence technologique estivale, Quebec City, QC, June, 2000

Norval, G.W., M.Stell, and F. Hancock, “Treatment of Spent Caustic Streams Using the ACCENT™ Process” 15th Canadian Symposium on Catalysis, Quebec City, Quebec, May 1998.

C.M. Evans and G.W. Norval, "Pilot Plant Requirements for Sulfuric Acid Processing", A.I.Ch.E. Spring National Meeting, Atlanta, GA, April 1994.

C.M. Evans, E.G. Lyne and G.W. Norval, "Treatment of Weak Acid Effluent from Metallurgical Acid Plant Gas Cleaning Systems", International Symposium on Drying, Roasting, Calcining and Acid Plant Design and Operation, Quebec City, Quebec, August 1993.

Norval, G.W., Missen, R.W., Smith, W.R., "Application of Concept of Equilibrium Constraints to Treatment of Aqueous Process Streams", A.I.Ch.E. 1992 National Meeting, Miami Beach, FL, November 1992.

Norval, G.W., and M.J. Phillips, "Application of Equilibrium Analysis to a Fischer-Tropsch Product", 11th Canadian Symposium on Catalysis, Halifax, NS, July 1990.

Norval, G.W., R.W. Missen and W.R. Smith, "Identification and Application of Partial Equilibrium", 40th Canadian Chemical Engineering Conference, Halifax, NS, July 1990.

Norval, G.W., and M.J. Phillips, "Mossbauer Spectroscopy Study of Iron Ammonia Synthesis Catalysts", 190th Meeting of the American Chemical Society, Chicago, IL, September 1985.

Poster

R. Dufresne, L. Caouette, G.W. Norval and C.J. Kanters, "Treatment of Clean Condensate using Catalytically Enhance Oxidation", TAPPI International Environment Meeting, Denver, CO, May 2000.

Appendix B

Claim 1	Claim 6
A method of producing soda ash and calcium chloride, comprising the steps of:	A method of producing soda ash and calcium chloride, comprising the steps of:
providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine;	providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine;
heating limestone to produce calcium oxide and carbon dioxide;	heating limestone to produce calcium oxide and carbon dioxide;
reacting the ammoniated brine with the carbon dioxide produced by the step of heating the limestone to produce sodium bicarbonate, ammonium chloride and a brine effluent;	reacting the ammoniated brine with the carbon dioxide produced by the step of heating the limestone to produce sodium bicarbonate, ammonium chloride and a brine effluent;
calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide;	calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide;
reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia; <u>wherein said step of reacting the calcium oxide with the ammonium chloride and brine effluent comprises reacting dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone;</u>	reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia;
recycling the ammonia to be used in the step of ammoniating the brine; and	recycling the ammonia to be used in the step of ammoniating the brine; and

recycling the water from the brine effluent to be used in the step of ammoniating the brine by crystallizing sodium chloride from the mixture of calcium chloride and brine effluent, separating the crystallized sodium chloride from the effluent by centrifuging the effluent, and recycling the crystallized sodium chloride;	recycling the water from the brine effluent to be used in the step of ammoniating the brine by crystallizing sodium chloride from the mixture of calcium chloride and brine effluent, separating the crystallized sodium chloride from the effluent by centrifuging the effluent, and recycling the crystallized sodium chloride;
evaporating any water remaining to produce water vapor;	evaporating any water remaining to produce water vapor;
recovering the produced calcium chloride;	recovering the produced calcium chloride;
recycling the water vapor and condensing into water;	recycling the water vapor and condensing into water;
wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method;	wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method;
collecting the produced calcium chloride;	collecting the produced calcium chloride;
separating residual salt; and	separating residual salt; and
forming an approximately 77 wt % solution of calcium chloride flakes.	forming an approximately 95 wt % solution of calcium chloride granules.