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

PETITION FOR *INTER PARTES* REVIEW OF U.S. PATENT NO. 8,591,852

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Citations to exhibits including patents, patent applications, and expert declarations reference original page, column, paragraph, or line numbers found in the underlying document. Citations to other exhibits refer to exhibit page numbering.

LIST OF CLAIMS

Claim 1:

1[Pre]	A method of producing soda ash and calcium chloride, comprising the steps of:
1[a]	providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine;
1[b]	heating limestone to produce calcium oxide and carbon dioxide;
1[c]	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;
1[d]	calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide;
1[e]	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;
1[f]	recycling the ammonia to be used in the step of ammoniating the brine; and
1[g]	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;
1[h]	evaporating any water remaining to produce water vapor;
1[i]	recovering the produced calcium chloride;
1[j]	recycling the water vapor and condensing into water;
1[k]	wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method;
1[l]	collecting the produced calcium chloride;
1[m]	separating residual salt; and
1[n]	forming an approximately 77 wt % solution of calcium chloride flakes.

Claim 2:

2	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.

Claim 3:

3	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.

Claim 4:

4	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.

Claim 5:

5[Pre]	A method of producing soda ash and calcium chloride, comprising the steps of:
5[a]	mixing sodium chloride and water to form fresh brine; providing ammonia gas; bubbling the ammonia gas through the fresh brine to form ammoniated brine;
5[b]	heating limestone in a lime kiln to produce dry calcium oxide and carbon dioxide;
5[c]	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;
5[d]	filtering the brine effluent to remove the precipitated sodium bicarbonate;
5[e]	calcining the filtered sodium bicarbonate at a temperature sufficient to decompose the sodium bicarbonate to form soda ash as a major product;

5[f]	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;
5[g]	recycling the ammonia gas produced by the reacting step to form additional ammoniated brine using the bubbling ammonia step;
5[h]	crystallizing sodium chloride in the brine effluent; centrifuging the brine effluent to separate the crystallized sodium chloride from the brine effluent;
5[i]	recycling the separated crystallized sodium chloride to form additional fresh brine using the mixing step;
5[j]	evaporating water from the effluent to form water vapor and to recover calcium chloride as a major product;
5[k]	collecting the produced calcium chloride;
5[l]	separating residual salt; and
5[m]	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;
5[n]	recycling the water vapor to form addition fresh brine using the mixing step;
5[o]	whereby all reactants are recycled, leaving substantially no waste to discharge.

Claim 6:

6[Pre]	A method of producing soda ash and calcium chloride, comprising the steps of:
6[a]	providing a source of brine; providing a source of gaseous ammonia; ammoniating brine with gaseous ammonia to form ammoniated brine;
6[b]	heating limestone to produce calcium oxide and carbon dioxide;
6[c]	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;
6[d]	calcining the sodium bicarbonate to decompose the sodium bicarbonate to produce soda ash and gaseous carbon dioxide;

6[e]	reacting the calcium oxide with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia;
6[f]	recycling the ammonia to be used in the step of ammoniating the brine; and
6[g]	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;
6[h]	evaporating any water remaining to produce water vapor;
6[i]	recovering the produced calcium chloride;
6[j]	recycling the water vapor and condensing into water;
6[k]	wherein the crystallized sodium chloride is further used for the providing of fresh brine by combining with the condensed water to continue the method;
6[l]	collecting the produced calcium chloride;
6[m]	separating residual salt; and
6[n]	forming an approximately 95 wt % solution of calcium chloride granules.

Claim 7:

7	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.

Claim 8:

8	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.

Claim 9:

9	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.

Claim 10:

10	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.

I. MANDATORY NOTICES

Real Party in Interest: China Chengda Engineering Co., Ltd. is the petitioner and a real party-in-interest. No other party was involved in any aspect of this Petition.

Related Matters: Petitioner is not aware of any pending matters related to the '852 Patent.

Lead Counsel and Back-Up Counsel: Petitioner designate lead and back-up counsel as noted below. A Power of Attorney is filed concurrently herewith under 37 C.F.R. § 42.10(b).

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II. REGULATORY REQUIREMENTS

Petitioner certifies under 37 C.F.R. § 42.104(a) that the '852 Patent is eligible for *inter partes* review and that Petitioner is not barred or estopped from requesting *inter partes* review on the grounds set forth herein.

III. OVERVIEW OF THE '852 PATENT

A. Background

1. Introduction

Sodium carbonate, more commonly known as soda ash, is one of the largest volume inorganic chemicals produced globally. EX1002,¶26. The Solvay process has been used for its manufacture since the 1860s and continues to be used today. *Id.*; EX1010, p.6. The Solvay process is well documented in decades-old or even century-old publications. EX1002,¶27 (citing EX1006 and EX1013).

The process may be summarized by the theoretical global equation involving the two main components: sodium chloride (NaCl) and calcium carbonate. EX1002,¶28.

$$2NaCl + CaCO_3 \rightarrow Na_2CO_3 + CaCl_2$$

The commercial success of the Solvay process owes largely to 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. EX1010, p.6. The brine absorbs carbon dioxide (CO_2), and a precipitate of sodium bicarbonate ($NaHCO_3$) is recovered. *Id*., p.7. Because CO_2 is an acidic gas, the system pH needs to be controlled for the production of sodium bicarbonate precipitate. EX1002,¶29. This is achieved through first buffering the brine solution with ammonia (NH_3), a weak base, to form ammoniated brine prior to CO_2 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; EX1013, p.98. Solvay process is thus also referred to as ammonia soda process. EX1002,¶29.

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 CO_2. EX1002,¶30. The latter is routinely recycled to the carbonation process. *Id*. (citing EX1010, p.7).

The Solvay process adds lime or milk of lime to recover ammonia from the process. EX1002,¶31. Lime (CaO), also known as quicklime, is obtained by calcination of limestone, which 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. EX1002,¶31.

CaO reacts with water and forms $Ca(OH)_2$, a base. *Id.*,¶32. 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. *Id.* The ammonia is then returned to the brine through absorption. *Id.*

The above ammonia-soda chemistry processes and related equipment were developed in the 1860s and those fundamentals have remained unchanged. *Id.*,¶33.

2. Using Dry Lime Without Slaking for Ammonia Recovery

Regarding recovery of ammonia from the waste effluent, adding dry lime directly into the effluent or adding 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. *Id.*,¶34; 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 (same); EX1010, p.8 ("In some operations, dry lime is used to produce a more concentrated calcium chloride by-product stream.").

Advantages of using dry lime instead of milk of lime have long been recognized. EX1002,¶35; EX1007, 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[.]"); *see also id*., 2:84-106 (listing advantages of using dry lime). Moreover, processes to use dry lime in Solvay processes have also been well-established. EX1002,¶35; EX1007, 2:118-4:25; EX1005, [0040].

3. Waste Recycling

The overall chemistry of the Solvay process can be written as follows:

$$2NaCl + CaCO_3 \rightarrow Na_2CO_3 + CaCl_2.$$

EX1002,¶36. Other reactants include ammonia, carbon dioxide, and water. *Id.*,¶37. POSITAs have long recognized that it is desirable to recycle these reactants as much as possible. *Id.; see also* EX1005, Abstract (characterizing an objective of the patent as "guaranteeing the recovery and recycling of ammonia and carbon dioxide), [0011], [0020]; 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").

Regarding 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. EX1002,¶38. 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. POSITAs have long understood that sodium chloride solubility decreases as the concentration of the

calcium chloride increases when water is removed through evaporation. EX1002, ¶38; EX1008, p.35 ("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):

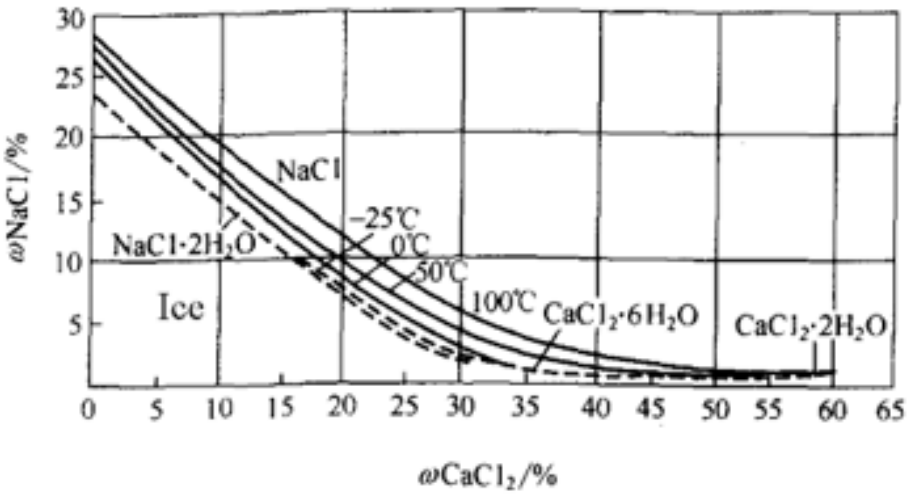


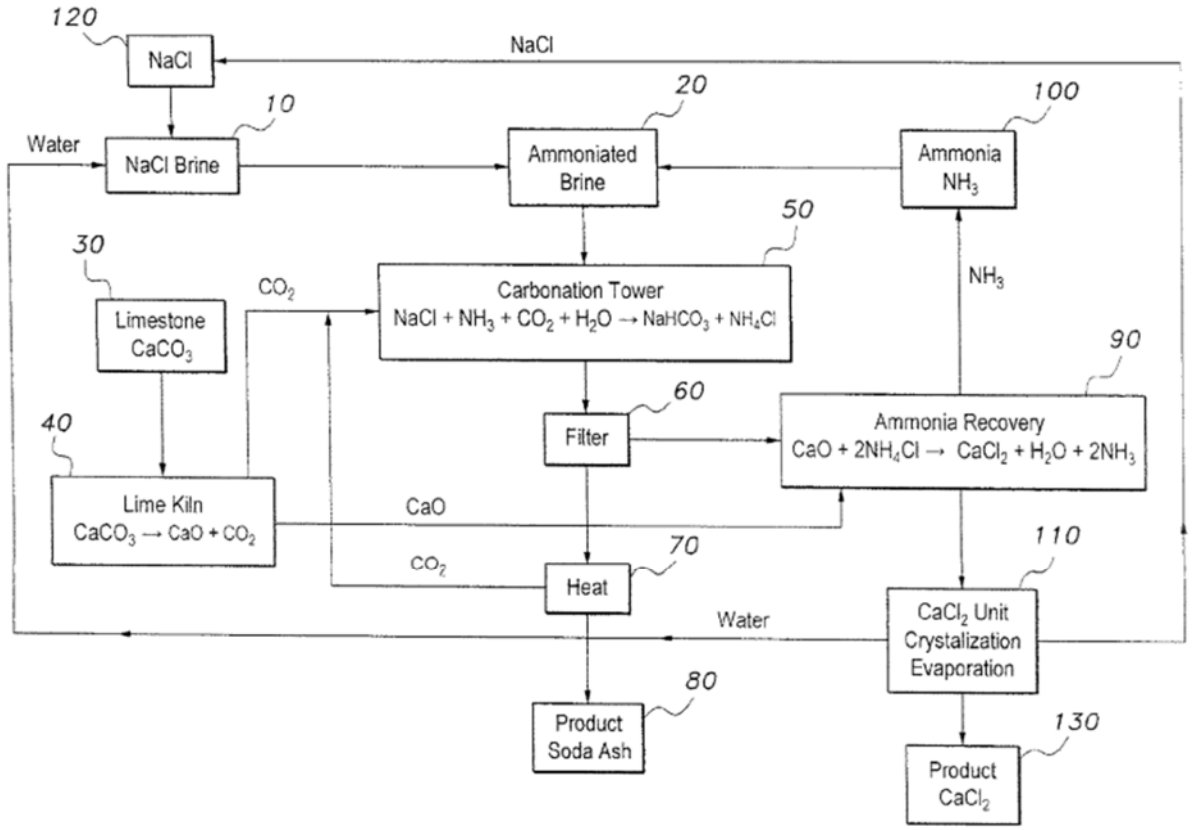
Fig. 16-4 Co-solubility of NaCl and $CaCl_2$ in water

Similarly, equipment used for water and salt recovery is also well-known in the art. EX1002,¶39. Accordingly, processes and equipment needed to recover various reactants are well known in the art. *Id*.

B. The ’852 Patent[1]

The ’852 Patent involves a method of producing soda ash and calcium chloride. EX1001, 1:13-17. The ’852 Patent admits 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 sole drawing of the ’852 Patent summarizes the claimed processes:

[1] Provided for background. Nothing herein is an admission that the ’852 Patent claims are adequately described or enabled.



EX1001, Figure.

The detailed description section of the ’852 Patent consists of mere 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,” all of the methods of producing soda ash and calcium chloride with minimal waste mentioned in the ’852 Patent, were well-known before the alleged invention of the ’852 Patent. EX1002,¶23.

C. Prosecution History

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. 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 (File History), p.74-81. In a June 12, 2013 office action, the Examiner again rejected the pending claims under 35 U.S.C. §112(b) and §103. *Id.*, p.90. 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. Thereafter, the Examiner issued a Notice of Allowance. *Id.*, p.108. However, the Examiner did not consider the prior art relied upon herein for the grounds of invalidity. *See* EX1001, References Cited.

IV. SUMMARY OF PRIOR ART

The '852 Patent claims priority to February 22, 2012, and is subject to the pre-AIA provision of 35 U.S.C. § 103.

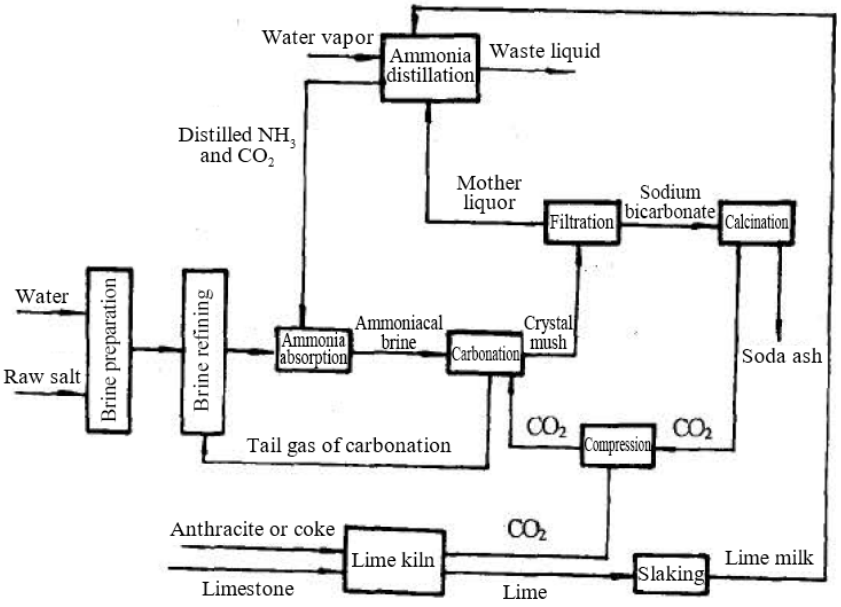
None of the references described below were considered during examination of the '852 Patent. EX1002,¶¶44-57.

A. *Fu* (EX1003)

Fu was published in September 1999. EX1003. *Fu* is prior art under pre-AIA

35 U.S.C. §§ 102(a) and (b). EX1015(Hall-Ellis Decl),¶¶44-50.

Fu describes a method of "Preparation of Soda Ash by ammonia soda Process." EX1003, p.50; EX1002,¶¶44-46. Figure 7-1 of *Fu* illustrates a flow diagram of the production process.



EX1003, Fig. 7-1. *Fu* discloses that the process for producing soda ash also comprises processing clarified liquid to produce calcium chloride. *Id*., p.76. *Fu* recognizes that this production process may generate waste materials or harmful substances that are not environmentally friendly. EX1003, p.75-76. *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*; EX1002,¶¶45-46.

B. *Xi* (EX1004)

Xi was published in 1990. EX1004. *Xi* is prior art under pre-AIA 35 U.S.C. §§ 102(a) and (b). EX1015,¶¶51-57.

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. *Id.*

C. *Du* (EX1005)

Du (CN 102,145,912) was issued on August 10, 2011. EX1005. *Du* is prior art under at least Pre-AIA 35 U.S.C. §102(a).

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

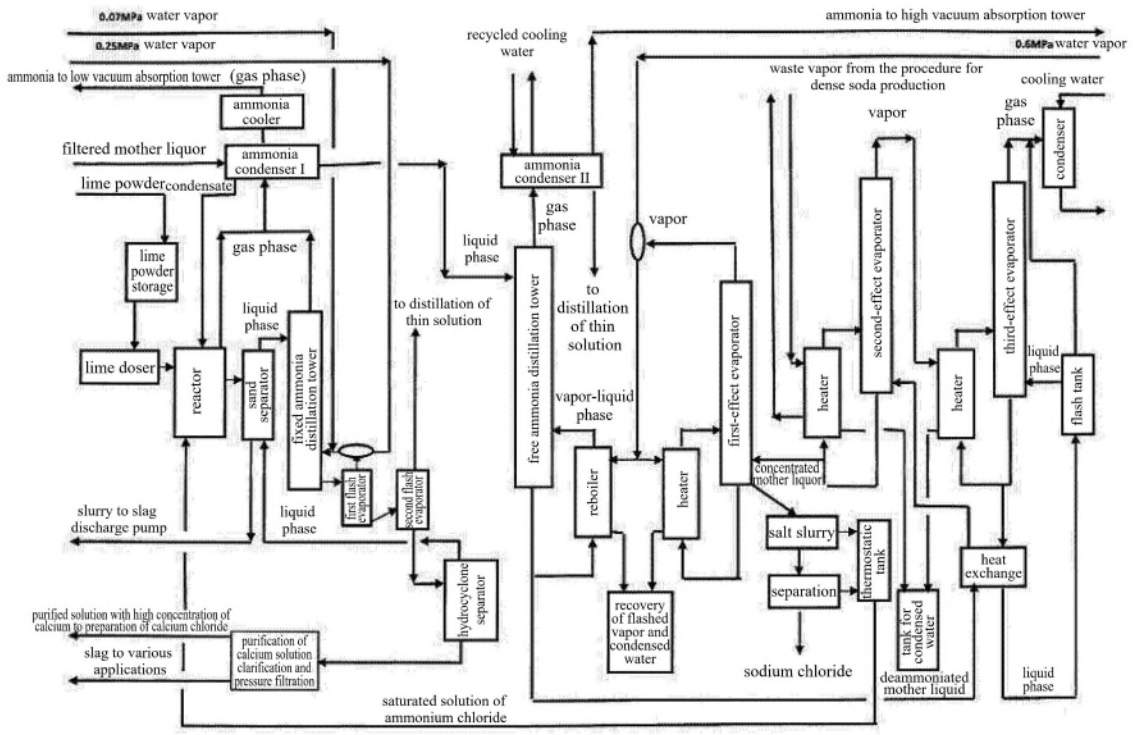


FIG. 1

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 chloride compound while guaranteeing the recovery and recycling of ammonia and carbon dioxide. *Id.*

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].

D. *Hou* (EX1006)

Hou was published at least by 1990. EX1006. *Hou* is prior art under pre-AIA 35 U.S.C. §§ 102(a) and (b). EX1015,¶¶58-64.

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.*

E. *Mohler* (EX1007)

Mohler was issued on June 12, 1934. EX1007. *Mohler* is prior art under pre-AIA 35 U.S.C. §§ 102(a), (b), and (e).

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 obtained in the Solvay process. *Id.*, 1:6-8. *Mohler* discloses various advantages of using dry quick lime to free the fixed ammonia (*id.*, 1:84-106) and teaches methods where dry lime can be introduced into the system against pressure through a conduit in finely divided form (*id.*, 2:1-9).

V. SUMMARY OF GROUNDS

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

VI. LEVEL OF ORDINARY SKILL

A person of ordinary skill in the art ("POSITA") at the time 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. EX1002,¶41.

VII. CLAIM CONSTRUCTION

Claim terms are construed according to their ordinary and customary meaning as understood by a POSITA and the prosecution history pertaining to the patent. 37

C.F.R. § 42.100(b). For purposes of this Petition, Petitioner interprets all claim terms in accordance with their ordinary and customary meaning.

VIII. INSTITUTION IS PROPER

A. Institution is Proper Under 35 U.S.C. § 314(a)

This is the first petition challenging the ’852 Patent. The *General Plastic* factors therefore do not provide a basis for denying institution.

Discretionary denial is also not applicable under the *Fintiv* factors because there is no pending litigation matter related to the ’852 Patent.

B. Institution is Proper Under 35 U.S.C. § 325(d)

None of the *Becton, Dickinson* factors weigh in favor of discretionary denial under 35 U.S.C. § 325(d). *See Becton, Dickinson & Co. v. B. Braun Melsungen AG*, IPR2017-01586, Paper 8 (Dec. 15, 2017) (precedential). None of the references relied upon by this Petition were before the Examiner, much less included in a rejection, during prosecution. Thus “the same or substantially the same prior art or arguments” were ***not*** previously presented to the Office.

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

A. Motivation to Combine

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 way 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. *Fu* discloses several examples that reduce water consumption. EX1002,¶60; *see also, e.g.*, EX1003, p.60 (improving crystal quality to reduce the amount of washing water), p.65 (maintaining proper temperature), p.79 (recycling "condensed water generated in the evaporators" and sending the condensed water to "a brine preparation field").

Similarly, *Xi* discloses methods to utilize and treat distillation waste liquor and waste residue from the soda ash production process. EX1002,¶61. 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." EX1004, p.19.

A POSITA would have been motivated to combine the soda ash and calcium chloride production process of *Fu* with the teachings of *Xi* because 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* provides an improvement to achieve that goal by using the dry quicklime without slaking to reduce water usage. EX1004, p.19; EX1002,¶62.

Accordingly, a POSITA would have been motivated to implement *Xi*'s use of quicklime instead of milk of lime used in *Fu*'s soda ash production process to further reduce water consumption of *Fu* in the reaction with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. EX1002,¶63. 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. *KSR Int'l Co. v. Teleflex Inc*., 127 S.Ct. 1727, 1740 (2007); EX1002,¶63; *supra* Section III.A.2. Moreover, 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. EX1002,¶63. 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*. *Id.* Any equipment needed for such process adjustment would have been well-known in the art. *Id.; see also, 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.

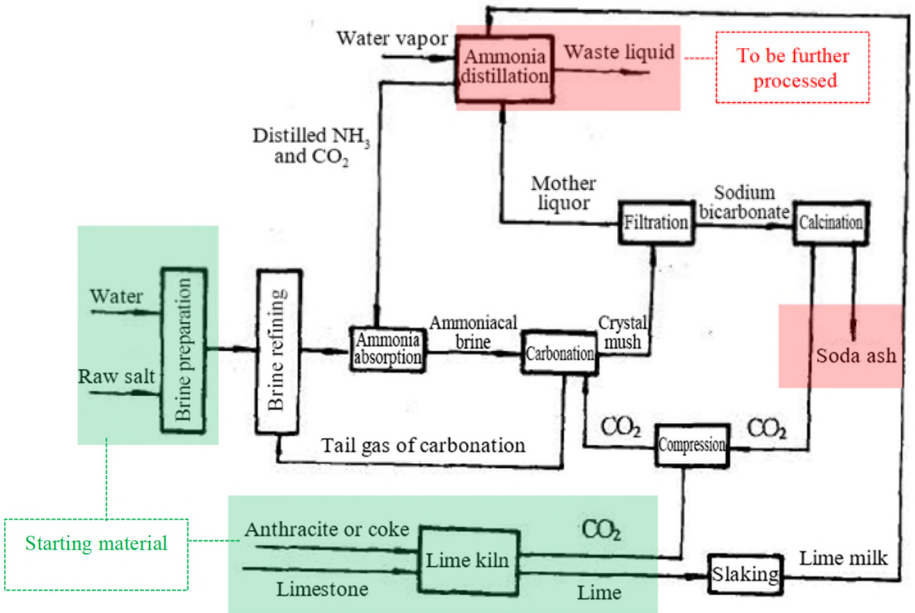
B. Claim 1

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

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:

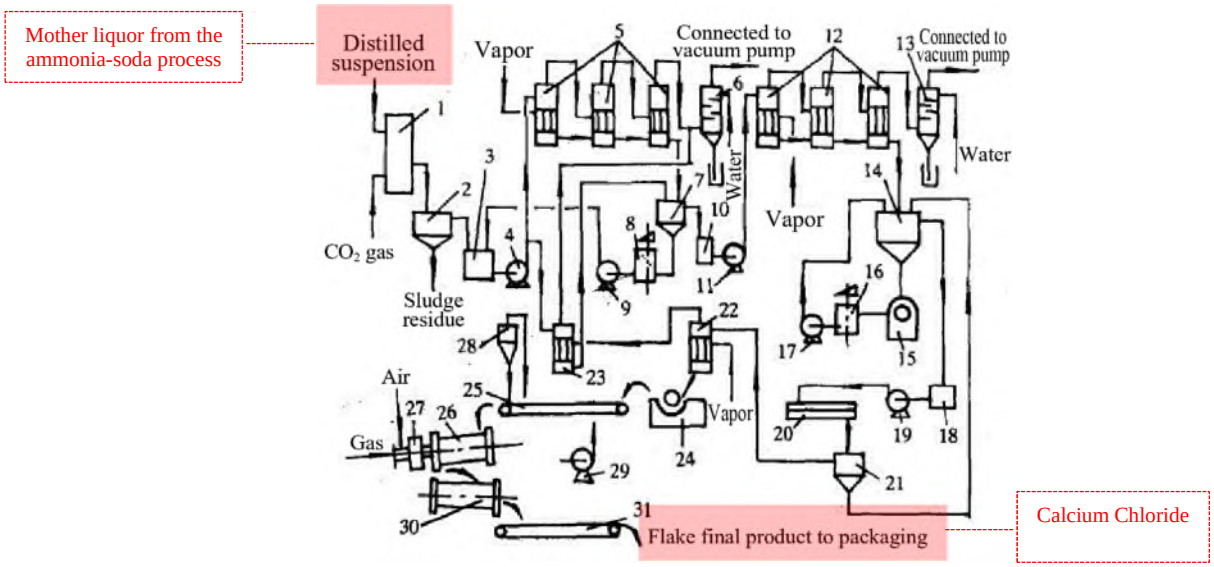
$$2NaCl+CaCO_3===Na_2CO_3+CaCl_2 \qquad (7\text{-}1)"$$

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); EX1002,¶64.

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



EX1003, Figure 7-25 (annotated); EX1002,¶65.

Accordingly, *Fu* discloses element 1[pre]. EX1002,¶¶64-66.

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

Fu discloses limitation [1a]. EX1002, ¶¶67-68. *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. …The ***brine*** enters an ammonia absorption tower to absorb the required amount of NH_3.")[2].

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

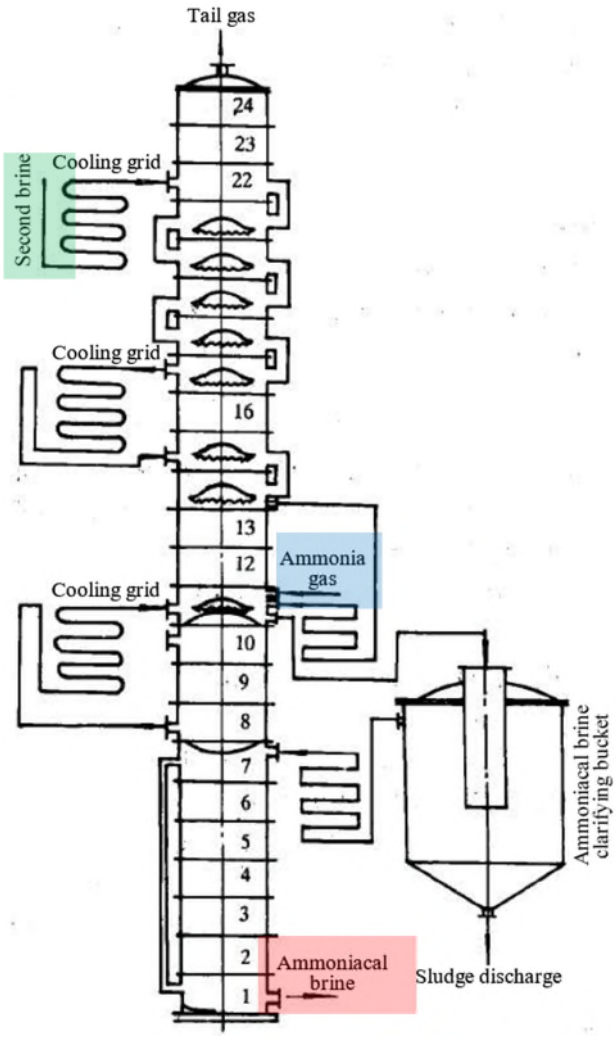
> Basic principle of ammonia absorption process: Ammonia absorption reaction and equilibrium partial pressure of gas above ammoniacal brine. The refined brine and the gas from ammonia distillation are mainly reacted as follows:
>
> $$NH_3(g) + H_2O(l) = NH_4OH(aq) + 35.2\ kJ/mol \quad (7\text{-}20)$$
> $$2NH_3(aq) + CO_2(g) + H_2O = (NH_4)_2CO_3(aq) + 95.0\ kJ/mol \quad (7\text{-}21)$$

EX1003, p.57;

> The brine enters an ammonia absorption tower to absorb the required amount of NH_3 and also absorbs a small amount of CO_2 at the same time.
>
> $$NH_3 + H_2O = NH_4OH + Q \quad (7\text{-}4)$$
> $$2NH_4OH + CO_2 = (NH_4)_2CO_3 + H_2O + Q \quad (7\text{-}5)$$

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

[2] Emphasis added unless otherwise noted.



EX1003, Figure 7-7 (showing the production of ammoniated brine with gaseous ammonia using an ammonia absorption tower); EX1002,¶68.

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

Fu discloses limitation [1b]. EX1002,¶69. *Fu* discloses heating limestone to produce calcium oxide and carbon dioxide. EX1003, p.52 ("The carbonation process requires a large amount of CO_2 gas, such that limestone is calcined to produce CO_2

and lime."), p.53 ("Practice has proved that the temperature of burning limestone is generally controlled within a range of 940-1200°C.").

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;"

Fu discloses limitation [1c]. EX1002,¶¶70-71. *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 CO_2 is introduced.
>
> $2NH_4OH+CO_2===(NH_4)_2CO_3+H_2O+Q$ (7-6)
>
> $(NH_4)_2CO_3+CO_2+H_2O===2NH_4HCO_3+Q$ (7-7)
>
> $2NH_4HCO_3+2NaCl===2NaHCO_3+2NH_4Cl+Q$ (7-8)

EX1003, p.51. After the carbonation process and precipitation of sodium bicarbonate, a brine effluent (i.e., the salt-containing liquid waste) is produced as well. EX1002,¶70; EX1003, p.52 ("the precipitation amount of $NaHCO_3$ 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.").

Fu further discloses at least some of the carbon dioxide reacting with the ammoniated brine is produced by the step of heating the limestone. EX1002,¶71; EX1003, p.52 ("The remaining CO_2 [required to form $NaHCO_3$] comes from a lime kiln. Calcium carbonate is decomposed into CO_2 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;"

Fu discloses limitation [1d]. EX1002,¶72. *Fu* teaches, "A filter cake containing $NaHCO_3$ is calcined to obtain final products Na_2CO_3 and CO_2." 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"

Fu alone or further in view of *Xi* renders limitation [1e] obvious. EX1002,¶¶73-77. 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 IX.B.iv. *Fu* further discloses producing calcium chloride, brine effluent, and ammonia by reacting calcium hydroxide with ammonium chloride and brine effluent. EX1002,¶73; EX1003, p.52 ("The ammonia is driven out by steam in a gas-liquid counterflow device. … The reaction is as follows: $2NH_4Cl+Ca(OH)_2===2NH_3+CaCl_2+2H_2O$ (7-12)").

A POSITA would have found it an obvious process choice, in view of *Fu*, to "react[] dry calcium oxide with the ammonium chloride and brine effluent without slaking the calcium oxide produced by heating the limestone." EX1002,¶74. First,

Fu discloses heating limestone to produce calcium oxide and carbon dioxide. EX1003, p.52 ("Calcium carbonate is decomposed into CO_2 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."). Second, 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. EX1002,¶74. Whether dry calcium oxide is added to the liquid effluent with or without first slaking the calcium oxide, the chemical reactions remain the same. *Id.* A POSITA would understand that this modification would not interfere with the other aspects of *Fu*'s processes. *Id.*

In addition, a POSITA would understand that slaking the calcium oxide requires a separate step of adding water to the dry calcium oxide. *Id.*,¶75. It would have been an obvious variation for a POSITA to add calcium oxide directly to the ammonium chloride and brine effluent instead of carrying out a separate step to first slake the calcium oxide with water. *Id.*; *see also, 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 (same); EX1010, p.8 ("In some operations, dry lime is used to produce a more concentrated calcium chloride by-product stream."). Moreover, 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. EX1002,¶75.

Alternatively, as described in Section IX.A, 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, thereby reducing water consumption and thus waste—a common goal of both *Fu* and *Xi*. EX1002,¶76; EX1004, p.19.

Accordingly, *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"

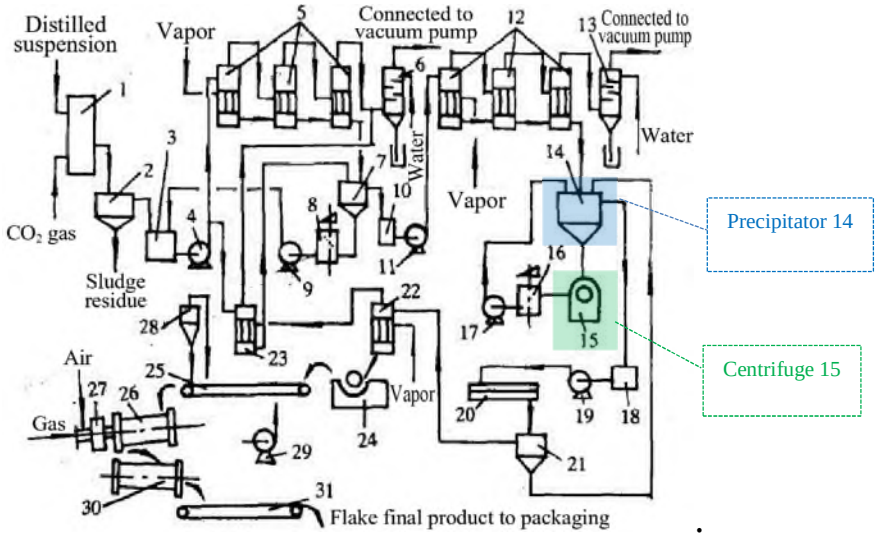
Fu discloses limitation [1f]. EX1002,¶78. *Fu* teaches that the ammonia is continuously recycled because it is "more expensive" and "only acts as a medium here." EX1003, p.51. To achieve that goal, "[t]he NH_3 in the mother liquor must be distilled out and returned for absorption." *Id.*, p.52. *Fu* 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;"

Fu discloses limitation [1g]. EX1002,¶¶79-81. *Fu* discloses that sodium chloride is crystalized and separated from the mixture of calcium chloride and brine effluent by concentrating the liquid through evaporation. *Id*.,¶79; 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% $CaCl_2$. … The partially concentrated and deseeded distillate is … concentrated to contain 38% calcium chloride and ***a main part of sodium chloride is separated***.").

Fu further discloses that the crystallized sodium chloride is separated from the effluent by centrifuging the effluent and recycled for soda ash production. EX1002,¶80; EX1003, 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."), Figure 7-25 (showing a process diagram of preparing calcium chloride and simultaneously separating NaCl):



Fu further teaches recycling water from the brine effluent by evaporation and condensation to be used in the step of ammoniating the brine.[3] EX1002, ¶80; EX1003, p.79 ("The amount of condensed water generated … is sent to a brine preparation field, and thus the water consumption can be appropriately reduced.").

[3] A POSITA would have understood, in view of the specification, that the step of ammoniating the brine includes the brine preparation step. EX1002,¶81, n.1. Indeed, the '852 Patent only mentions a single purpose of the recycled water, that is to "make more brine." EX1001, 2:64-66. Thus, "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. EX1002,¶81.

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

Fu discloses limitation [1h]. EX1002,¶82. *Fu* discloses "[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% $CaCl_2$" and forming "flakes containing the main substance of about 78% $CaCl_2$." EX1003, p.79. A POSITA would understand when the solution is "concentrated," water is evaporated from the solution in the evaporator. EX1002,¶82. A POSITA would also understand it is not practically possible, nor necessary, to evaporate ***all*** water remaining. *Id.* 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 ($CaCl_2$)[.]"). Accordingly, 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. EX1002,¶82.

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

Fu discloses limitation [1i]. EX1002,¶83. *Fu* teaches that the produced calcium chloride is recovered. EX1003, p.79 ("The 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.").

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

Fu discloses limitation [1j]. EX1002,¶84. *Fu* discloses that water vapor is generated in the evaporators and recycled. EX1003, p.79 ("The amount of condensed water generated in the evaporators ... 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.*; EX1002,¶84. *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% $CaCl_2$." EX1003, p.79. A POSITA would understand the condensed water "sent to a brine preparation field" contains water generated from all evaporators, including evaporator 22. EX1002,¶84.

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;"

Fu discloses or renders obvious limitation [1k]. EX1002,¶¶85-87. *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."). *Fu* also discloses "the condensed water is sent to a brine preparation field, and thus the water consumption can be appropriately reduced." *Id.*, p.79.

A POSITA would have understood that *Fu* teaches combining the crystalized sodium chloride and condensed water 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. EX1002,¶86.

Alternatively, it would have been obvious for a POSITA to mix the recycled sodium chloride and condensed water of *Fu* to provide the fresh brine needed in further soda ash production. *Id.*,¶87.

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

Fu discloses limitation [1l]. EX1002,¶88. *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"

Fu discloses limitation [1m]. EX1002,¶¶89-91. As discussed above regarding limitation [1g], *Fu* discloses, "[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.

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"); *see also* EX1002,¶90.

Alternatively, 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. EX1002,¶91; *see also, 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 (disclosing similar steps to separate residual salt).

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

Fu discloses limitation [1n]. EX1002,¶92. *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."). A POSITA would understand that calcium chloride flakes containing 78% $CaCl_2$ satisfies the requirement of "approximately 77 wt % solution of calcium chloride flakes." EX1002,¶92. Further, a POSITA would have known

that a common form of calcium chloride hydrates is $CaCl_2 \cdot 2H_2O$, in which the weight percentage of calcium chloride is 75.5%. *Id.* (citing EX1014). Thus, it would also have been obvious to a POSITA to form an approximately 77 wt % solution of calcium chloride flakes. *Id.*

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."

Fu in view of *Xi* renders obvious claim 2. EX1002,¶¶93-94. 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 $NaHCO_3$ 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.

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."

Fu in view of *Xi* renders obvious claim 3. EX1002,¶95. 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, $NaHCO_3$ 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."

For the same reasons above regarding limitation [1k], *Fu* discloses this limitation. *See* Section IX.B.xii. Thus, *Fu* in view of *Xi* renders obvious claim 4. EX1002,¶96.

F. Claim 5

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

Fu disclosed a method of producing soda ash and calcium chloride for the same reasons above regarding [1Pre]. *See* Section IX.B.i; EX1002,¶97.

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;"

Fu discloses limitation [5a]. EX1002,¶98. *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 NH_3" and "[t]he ammonia absorption tower is a multi-section cast iron single bubble-cap tower." *Id.*, p.51, p.59. A POSITA would have understood that gas absorption is performed by bubbling gas through liquid and a multi-section cast iron bubble-cap tower is a typical bubbler used in the industry. EX1002,¶98; *see also* 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."); EX1016, 20.

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

Fu discloses limitation [5b] for the same reasons above regarding [1b]. *See* Section IX.B.iii; EX1002,¶99.

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;"

Fu discloses limitation [5c]. EX1002,¶¶100-102. *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 CO_2 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 when the carbon dioxide is introduced into the carbonation tower, it is bubbling through the ammoniated brine facilitated by the disclosed tower structure. EX1002,¶100; *see also* 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.").

Fu discloses the limitation "to produce sodium bicarbonate, ammonium chloride, and a brine effluent" for the same reasons above regarding [1c]. Section IX.B.iv; EX1002,¶101.

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."). Moreover, a POSITA would have understood

that the ammonium chloride produced during the carbonization process is soluble in the brine effluent. EX1002,¶102.

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

Fu discloses limitation [5d]. EX1002,¶103. *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;"

Fu discloses limitation [5e] for at least the same reasons discussed above regarding claim 3. Section IX.D; EX1002,¶104.

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;

Fu alone in view of the knowledge of a POSITA, or *Fu* in view of *Xi* renders obvious claim limitation [5f] for the same reasons above regarding [1e]. Section IX.B.vi; EX1002,¶105.

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

Fu discloses limitation [5g] for the same reasons above regarding [1f]. Section IX.B.vii; EX1002,¶106.

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

Fu discloses limitation [5h] for the same reasons above regarding [1g]. Section IX.B.viii; EX1002,¶107.

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

Fu discloses or renders obvious limitation 5[i] for the same reasons above regarding [1k]. Section IX.B.xii; EX1002,¶108.

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

Fu discloses limitation [5j] for the same reasons above regarding [1h] and [1i]. Sections IX.B.ix-x; EX1002,¶109.

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

Fu discloses limitation [5k] for the same reasons above regarding [1l]. Section IX.B.x; EX1002,¶110.

xiii. [5l] "separating residual salt; and"

Fu discloses or renders limitation [5l] obvious for the same reasons above regarding [1m]. Section IX.B.xiv; EX1002,¶111.

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."

Fu discloses limitation [5m] for at least the same reasons above regard limitation [1n]. Section IX.B.xv; EX1002,¶112; *see also infra* Section IX.G.ii (limitation [6n]).

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

Fu discloses limitation [5n] for the same reasons above regarding [1j] and [1k]. Sections IX.B.xi-xii; EX1002,¶113.

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

Fu discloses or renders obvious limitation [5o]. EX1002,¶114. 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 IX.B.vii), sodium chloride (Sections IX.B.viii and IX.B.xiv), water (Sections IX.B.viii and IX.B.xi), and carbon dioxide (Section IX.B.iv). EX1002,¶114. Moreover, 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. *Id.* Indeed, nowhere in the '852 Patent specification mentions the recycling of calcium oxide. *Id.* Because all the remaining reactants are recycled, there is substantially no waste to discharge following the methods disclosed in *Fu*. *Id.*

Alternatively, 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. EX1002,¶115.

G. Claim 6

i. [6Pre]-[6m]

Fu discloses or renders obvious limitations [6Pre] to [6m] for the same reasons above regarding identical or similar limitations [1Pre] to [1m]. EX1002,¶116; *id*., Appendix B; Sections IX.B.i-xiv.

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

Fu renders obvious limitation [6n]. EX1002,¶117. As discussed above regarding limitation 1[n], *Fu* discloses forming an approximately 77 wt % solution of calcium chloride flakes. Section IX.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. EX1002,¶117 (citing EX1014, p.4). It would have been obvious to a POSITA to heat the 77 wt% solution of calcium

chloride to further remove the water until an approximately 95 wt% solution of calcium chloride granules (anhydrous calcium chloride) was obtained. EX1002,¶117. 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 calcium chloride products. *Id*.; *see also* EX1014 (showing different calcium chloride product offerings). Moreover, 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. EX1002,¶117. Accordingly, *Fu* in view of *Xi* renders claim 6 obvious. *Id.*

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."

Claim 7 depends on claim 6 and recites the same claim limitation as in claim 2. Accordingly, *Fu* in view of *Xi* renders obvious claim 7 for the same reasons above regarding claims 2 and 6. *See* Sections IX.C and IX.G; EX1002,¶118.

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."

Claim 8 depends on claim 7 and recites the same claim limitation as in claim 3. Accordingly, *Fu* in view of *Xi* renders obvious claim 8 for the same reasons above regarding claims 3 and 7. *See* Sections IX.D and IX.H; EX1002,¶119.

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."

Claim 9 depends on claim 6 and recites the same claim limitation as in claim 4. Accordingly, *Fu* in view of *Xi* renders obvious claim 9 for the same reasons above regarding claims 4 and 6. *See* Sections IX.E and IX.G; EX1002,¶120.

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."

Claim 10 depends on claim 6 and recites the claim limitation similar to [1e]. Accordingly, *Fu* in view of *Xi* renders obvious claim 10 for the same reasons above regarding claim 6 and [1e]. *See* Sections IX.B.vi and IX.G; EX1002,¶121.

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

A. Motivation to Combine

As discussed in Sections IX.A-B, *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; EX1002,¶122. Both *Fu* and *Hou* are directed to soda ash production and address similar problem related to the use of waste liquor. EX1002,¶122.

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. Because calcium chloride flakes and anhydrous calcium chloride granules are two common

forms of calcium chloride products, 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. EX1002,¶123. Moreover, 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*. *Id.*

B. Claim 6

i. [6Pre]-[6m]

Fu disclose or renders obvious limitations [6Pre] to [6m] for the same reasons above regarding identical or similar limitations [1Pre] to [1m]. *See* Section IX.B.i-xiv; EX1002,¶124.

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

As discussed above, it would have been obvious for a POSITA to combine *Fu* and *Hou*. Section X.A. *Fu* discloses a method of producing a 78% solution of calcium chloride. *See, e.g.*, Section IX.B.xv; EX1003, p.79. *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 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*. EX1002,¶125.

Accordingly, *Fu* in view of *Hou* renders obvious claim 6. *Id.*,¶¶124-26.

C. Claims 7-9

Fu in view of *Hou* renders obvious claims 7-9. EX1002,¶127. 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 IX.G-J.

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

As discussed above, 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. EX1002,¶128. A POSITA would have been similarly motivated to combine *Fu* with *Xi* and further with *Hou* for the same reasons. *Id.*

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 IX.G and IX.K (claims 6 and 10); EX1002,¶129.

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

A. Motivation to Combine

As discussed in Section IX.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* EX1002,¶130; EX1003, p.75-76.

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, less waste, and lower cost. *Id.*, 1:84-106; EX1002,¶131.

Both *Fu* and *Mohler* are related to soda ash production and address similar problem in connection with waste reduction. EX1002,¶132. 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. EX1002,¶132. Moreover, *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. A POSITA would understand that

Mohler provides an improvement to achieve that goal by using the dry quicklime without slaking to reduce water consumption and waste generation. EX1002,¶132.

Therefore, with respect to *Fu*'s soda ash and calcium chloride production process, a POSITA would have been motivated to use dry quick lime as taught by *Mohler* instead of milk of lime to react with the ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. *Id.*,¶133. 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. *KSR Int'l Co.*, 127 S.Ct. at 1740; EX1002,¶133; *supra* Section III.A.2. 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 milk of lime and perform the same functions as intended by *Fu*. EX1002,¶133. Moreover, 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. *Id*.

B. Claim 1

i. [1Pre]-[1d]

Fu discloses claim limitations [1Pre]-[1d] for the same reasons above regarding Ground 1A, [1Pre]-[1d]. Section IX.B.i-v; EX1002,¶134.

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"

Fu renders obvious limitation [1e] for the same reasons above. Section IX.B.vi.

Moreover, as described in Section XII.A, 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. EX1002,¶136. *Mohler* further discloses processes "for using lime in solid form in the recovery of ammonia from solution." EX1007, claim 5. *Mohler* also teaches that using solid lime in this process can reduce water consumption and waste generation. *See* Section XII.A. 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. EX1002,¶136.

Accordingly, 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. *Id.*,¶137. *Fu* in view of *Mohler* renders obvious limitation 1[e]. *Id.*

iii. [1f]-[1n]

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

Accordingly, *Fu* in view of *Mohler* renders obvious claim 1. EX1002,¶¶138-39.

C. Claim 2

Fu in view of *Mohler* renders obvious claim 2 for the same reasons above regarding claim 1 and claim 2 (Ground 1A). *See* Sections XII.B and IX.C; EX1002,¶140.

D. Claim 3

Fu in view of *Mohler* renders obvious claim 3 for the same reasons above regarding claim 2 and claim 3 (Ground 1A). *See* Sections XII.C and IX.D; EX1002,¶141.

E. Claim 4

Fu in view of *Mohler* renders obvious claim 4 for the same reasons above regarding claim 1 and claim 4 (Ground 1A). *See* Sections XII.B and IX.E; EX1002,¶142.

F. Claim 5

i. [5Pre]-[5e]

Fu discloses or renders obvious limitations [5Pre]-[5e] for the same reasons above regarding Ground 1A, limitations [5Pre]-[5e]. *See* Sections IX.F.i-vi; EX1002,¶143.

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;

Fu in view of *Mohler* renders obvious limitation [5f] for the same reasons above regarding [1e]. Section XII.B.ii.; EX1002,¶144.

iii. [5g]-[5o]

Fu in view of *Mohler* renders obvious claim limitations [5g]-[5o] for the same reasons above regarding Ground 1A, claim limitations [5g]-[5o]. Sections IX.F.viii-xvi; EX1002,¶145.

G. Claims 6-9

Fu in view of *Mohler* renders obvious claims 6-9 for the same reasons above regarding Ground 1A, claims 6-9. Section IX.G-J; EX1002,¶146.

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."

Claim 10 depends on claim 6 and further recites the claim limitation similar to [1e]. Accordingly, *Fu* view of *Mohler* renders obvious claim 10 for the same reasons above regarding claim 6 and [1e]. Sections IX.G. and XII.B.ii. EX1002,¶147.

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

As discussed above, a POSITA would have been motivated to combine the teachings of *Fu* with those of *Mohler* to reduce water consumption (Section XII.A) and to combine the teachings of *Fu* with those of *Hou* to prepare anhydrous $CaCl_2$ (Section X.A). 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. EX1002,¶148.

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. Sections XII.G and XI (claims 6 and 10); EX1002,¶149.

XIV. GROUND 2: *DU* IN VIEW OF *FU* AND *HOU* RENDERS OBVIOUS CLAIMS 1-10

A. Motivation to Combine

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]; EX1002,¶150.

As discussed in Section IX.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 IX.B.vii), sodium chloride (Sections IX.B.viii and IX.B.xiv), water (Sections IX.B.viii and IX.B.xi), and carbon dioxide (Section IX.B.iv).

Because *Fu* and *Du* are related to soda ash production and address similar problem in connection with waste reduction, 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. EX1002,¶152.

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].

Fu discloses reacting calcium hydroxide with ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. EX1002,¶154. 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. *Id*. 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. *Id*. 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*. *Id*.

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. EX1002,¶155; *KSR Int'l Co.*, 127 S.Ct. at 1740.

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). EX1002,¶156. 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. *Id.*

B. Claim 1

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

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].

Accordingly, *Du* discloses 1[pre]. EX1002,¶158.

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

Du discloses limitation [1a]. EX1002,¶159. *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;"

Du alone, or *Du* in view of *Fu*, renders obvious claim limitation [1b]. EX1002,¶¶160-62. *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].

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]. 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. EX1002,¶161; *see also* EX1005, [0017] (disclosing adding the lime powder into the reaction solution by "a crew conveyor"); EX1010, p.8.

Alternatively, *Fu* discloses heating limestone to produce calcium oxide and carbon dioxide. EX1003, p.52 ("The carbonation process requires a large amount of CO_2 gas, such that limestone is calcined to produce CO_2 and ***lime***."). 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*. EX1002,¶162.

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;"

Du alone, or *Du* in view of *Fu*, renders obvious limitation [1c]. EX1002 ¶¶163-64. 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."

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] (same).

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

Du discloses limitation [1d]. EX1002,¶165. *Du* teaches that "[t]he separated sodium bicarbonate is calcined into soda." EX1005, [0027]. A POSITA would have understood that when sodium bicarbonate is calcined, it will decompose and produce soda ash and gaseous carbon dioxide. EX1002, ¶165; *see also* EX1013, p.97; EX1010, p.8. Alternatively, *Du* in view of *Fu* renders limitation [1d] obvious for the reasons discussed above in Sections IX.B.v and XIV.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"

Du alone or further in view of *Fu* renders limitation [1e] obvious. EX1002,¶¶166-70. 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 XIV.B.iv.

Du further discloses producing calcium chloride and ammonia by reacting lime powder with ammonium chloride solution. EX1005, [0031] ("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***.").

As discussed above, a POSITA would have understood that lime powder produced by "grinding in a lime procedure" refers to dry calcium oxide powder. EX1002,¶168. Thus, *Du* discloses "said step of reacting the calcium oxide . . . without slaking the calcium oxide produced by heating the limestone." *See also* EX1005, [0040].

To the extent that 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, such an argument is incorrect. 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. EX1002,¶169; *see also* 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.***") Accordingly, when the ammonium chloride solution reacts with calcium oxide in *Du*, a POSITA would understand that the ammonium chloride solution also contains brine effluent, which will remain after the reaction. EX1002,¶169.

Alternatively and/or additionally, *Du* in view of *Fu* renders limitation [1e] obvious. EX1002,¶170. As discussed above in Section XIV.A, *Fu* discloses reacting calcium hydroxide with ammonium chloride and brine effluent to produce calcium chloride, brine effluent, and ammonia. 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. EX1002,¶170. 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. *Id*. 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*. *Id*. 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. *Id*.

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

Du discloses or renders obvious limitation [1f]. EX1002,¶171. *Du* teaches the disclosed invention "guarantee[s] the recovery and recycling of ammonia and carbon dioxide." *See* EX1005, Abstract, [0011], [0020]. A POSITA would have understood that the recycled ammonia would be reused in the step of ammoniating the brine. EX1002,¶171; *see also* Section III.A.3.

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;"

Du alone or further in view of *Fu* renders limitation [1g] obvious. EX1002,¶¶172-74. *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."). 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. EX1002, ¶172; *see also* EX1003, p.77-78; EX1013, p.68.

To the extent that PO asserts 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 XIV.B.vi; EX1002, ¶173. Because sodium chloride does not participate in the reaction of calcium oxide and ammonium chloride, a POSITA would have understood that the sodium chloride can be separated after the reaction without having any impact on the process. *Id.* And for the same reasons discussed above in Section XIV.A, 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. EX1002, ¶173.

Moreover, *Du* teaches "recycling the water from the brine effluent to be used in the step of ammoniating the brine." For example, *Du* discloses, "[d]uring 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 IX.B.viii; EX1002, ¶174.

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

Du discloses or renders obvious limitation [1h]. EX1002,¶175. *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 and/or additionally, 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*. EX1002,¶175; *See also* EX1005, claim 5; Section IX.B.ix.

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

Du discloses limitation [1i]. EX1002,¶176. *Du* teaches that the produced calcium chloride is recovered. *See*, *e.g*., EX1005, [0033].

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

Du discloses limitation [1j]. EX1002,¶177. *Du* discloses water vapor is generated in various 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;"

Du renders obvious limitation [1k]. EX1002,¶178. As discussed above regarding elements [1g] and [1j], *Du* discloses recovering crystallized sodium chloride, and recycling water by vaporization and condensation. Sections XIV.B.viii and XIV.B.xi. It would have been obvious for a POSITA to combine the crystallized sodium chloride with the condensed water to provide fresh brine and continue the

method. EX1002,¶178; 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 IX.B.xii and XIV.A. EX1002,¶178.

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

Du discloses limitation [1l]. EX1002,¶179. *Du* teaches collecting the produced calcium chloride in "the tank for thick calcium solution." EX1005, [0033].

xiv. [1m] "separating residual salt; and"

Du alone or further in view of *Fu* renders obvious limitation [1m]. EX1002,¶180. As discussed above, *Du* and *Fu* each discloses separating salt through evaporation and crystallization. Sections IX.B.viii, IX.B.xiv, XIV.B.viii. It would have been obvious for a POSITA to further separate residual salt from the calcium chloride product of *Du* to ensure the quality of the calcium chloride produced, at least in view of the express teaching of *Fu*. EX1002,¶180; Section IX.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."

Du alone, in view of *Fu,* or further in view of *Hou* renders obvious limitation [1n]. EX1002,¶¶181-82. *Du* discloses "achieving a concentration of calcium chloride being 30% (wt) or higher." EX1005, [0011]. 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. EX1002, ¶181 (citing EX1014, p.4). It would have been obvious to further concentrate calcium chloride of *Du* to arrive at an approximately 77 wt % solution of calcium chloride flakes. *Id*.

Moreover, *Fu* discloses forming calcium chloride flakes with approximately 77 wt% calcium chloride. EX1003, p.79; Section IX.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 $CaCl_2$•$2H_2O$." EX1006, p.15. A POSITA would understand that calcium chloride flakes containing 75% $CaCl_2$ discloses approximately 77 wt % solution of calcium chloride flakes. EX1002,¶182. 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, achieving the benefits of such a product described in *Fu* and/or *Hou*. *Id.*

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."

Du in view of *Fu* and *Hou* renders obvious claim 2. EX1002,¶183. 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."). Moreover, *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."

Du in view of *Fu* and *Hou* renders obvious claim 3. EX1002,¶184. Further to the discussion above regarding claims 1 and 2, *Du* teaches that "[t]he separated sodium bicarbonate is calcined into soda." EX1005, [0027]. Calcination of sodium bicarbonate is known to involve heating the sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate. EX1002,¶184; *see also* EX1003, p.68. Alternatively, it would have been obvious to heat the filtered sodium bicarbonate to a temperature sufficient to decompose the sodium bicarbonate, as taught by *Fu*. EX1002,¶184.

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."

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 XIV.B.xii, *Du* in view of *Fu* and *Hou* renders obvious claim 4. EX1002,¶185.

F. Claim 5

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

Du disclosed a method of producing soda ash and calcium chloride for the same reasons above regarding [1Pre]. *See* Section XIV.B.i EX1002,¶186.

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;"

Du discloses or renders obvious limitation [5a]. EX1002,¶187. *Du* discloses that "the ammonia must be absorbed [] with the sodium chloride solution" in an "ammonia absorption tower." EX1005, [0024]. 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. EX1002,¶187.

Alternatively, the combination of *Du* and *Fu* further discloses this limitation. EX1002,¶188. As discussed in Section IX.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;"

Du alone, or *Du* in view of *Fu*, renders obvious limitation [5b] for the same reasons above regarding [1b]. Section XIV.B.iii; EX1002,¶189.

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;"

Du discloses or renders obvious limitation [5c]. EX1002,¶¶190-91.

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]. 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. EX1002,¶190.

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"); EX1002,¶191.

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

Du discloses limitation [5d]. EX1002,¶192. *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;"

Du discloses limitation [5e] for at least the same reasons discussed above regarding claim 3. Section XIV.D; EX1002,¶193.

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;

Du alone, or *Du* in view of *Fu* renders obvious claim limitation [5f] for the same reasons above regarding [1e]. Section XIV.B.vi; EX1002,¶194.

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

Du discloses or renders obvious limitation [5g] for the same reasons above regarding [1f]. Section XIV.B.vii; EX1002,¶195.

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

Du discloses or renders obvious limitation [5h] for the same reasons above regarding [1g]. Section XIV.B.viii; EX1002,¶196.

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

Du renders obvious limitation [5i] for the same reasons above regarding [1k]. Section XIV.B.xii; EX1002,¶197.

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

Du discloses limitation [5j] for the same reasons above regarding [1h] and [1i]. Sections XIV.B.ix-x; EX1002,¶198.

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

Du discloses limitation [5k] for the same reasons above regarding [1l]. Section XIV.B.x; EX1002,¶199.

xiii. [5l] "separating residual salt; and"

Du discloses or renders limitation [5l] obvious for the same reasons above regarding [1m]. Section XIV.B.xiv; EX1002,¶200.

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."

Du in view of *Fu* and *Hou* renders obvious limitation [5m] for at least the same reasons above regard limitation [1n]. Section XIV.B.xv; EX1002,¶201; *see also infra* Section XIV.G.ii (limitation [6n]).

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

Du renders obvious limitation [5n] for the same reasons above regarding [1j] and [1k]. Sections XIV.B.xi-xii; EX1002,¶202.

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

Du in view of *Fu* renders obvious limitation [5o]. EX1002,¶¶203-04. 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. EX1002,¶203. *Du* also "guarantee[s] the recovery and recycling of ammonia and carbon dioxide." EX1005, Abstract. A POSITA would have understood that calcium oxide is typically consumed in the soda ash production process and incorporated in the final calcium chloride products, leaving little to recycle. EX1002,¶203. Because all the remaining reactants are recycled, there is

substantially no waste to discharge following the methods disclosed in *Fu* and/or *Du*. EX1002,¶203; *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.").

Alternatively, 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. EX1002,¶204.

G. Claim 6

i. [6Pre]-[6m]

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]. Sections XIV.B.i-xiv; EX1002,¶205, Appendix B.

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

Du discloses "achieving a concentration of calcium chloride being 30% (wt) or higher." EX1005, [0011]. A POSITA would have understood that concentrated calcium chloride in liquid form or in anhydrous form are common types of calcium chloride end products. EX1002,¶ 206; *See also* 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 of *Du* to arrive at an approximately 95 wt % solution of calcium chloride granules. EX1002,¶ 206.

Moreover, *Hou* expressly teaches preparation of 95% calcium chloride. EX1006, p.16. It would also have been obvious for a POSITA to combine *Du* with *Fu* and *Hou* to increase the concentration of calcium chloride to approximately 95 wt% by evaporating additional water from the calcium chloride solution as taught by *Hou*, achieving the benefits of such a product described in *Hou*. EX1002,¶207. Accordingly, *Du* in view of *Fu* and *Hou* renders obvious claim 6. EX1002,¶208.

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."

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. EX1002,¶209; Sections XIV.C and XIV.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."

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. EX1002,¶210; Sections XIV.D and XIV.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."

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. EX1002,¶211; Sections XIV.E and XIV.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."

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]. EX1002,¶212; Sections XIV.G and XIV.B.vi.

XV. SECONDARY CONSIDERATIONS

Petitioner is aware of none, and requests the opportunity to address any if raised by Patent Owner.

XVI. CONCLUSION

Petitioner respectfully request that IPR be instituted and the challenged claims be cancelled.

Respectfully submitted,
BAKER BOTTS L.L.P.

December 16, 2022
Date

/ Eliot D. Williams /
Eliot D. Williams (Reg. No. 50,822)
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CERTIFICATE OF COMPLIANCE

1. Pursuant to 37 C.F.R. §42.24(d), the undersigned certifies that the foregoing Petition, exclusive of the exempted portions as provided in 37 C.F.R. §42.24(a), contains 13,991 words which is no more than 14,000 words and therefore complies with the type-volume limitations of 37 C.F.R. §42.24(a). The word count was calculated by starting with Microsoft Word's total document word count and subtracting the words for the Table of Contents, the Exhibit List, Claims List, the Mandatory Notices, the Certificate of Compliance, and the Certificate of Service.

2. This Petition complies with the general format requirements of 37 C.F.R. § 42.6(a) and has been prepared using Microsoft® Word in 14-point Times New Roman.

Respectfully submitted,
BAKER BOTTS L.L.P.

December 16, 2022
Date

/ Eliot D. Williams /
Eliot D. Williams (Reg. No. 50,822)
LEAD COUNSEL FOR PETITIONER

CERTIFICATE OF SERVICE

In accordance with 37 C.F.R. §§42.6(e) and 42.105, the undersigned certifies that on the 16th day of December, 2022, a complete and entire copy of the PETITION FOR *INTER PARTES* REVIEW ("petition"), Petitioner' power of attorney, and related Exhibits were served on Patent Owner via Express Mail or by means at least as fast and reliable as Express Mail at the correspondence address of record for the subject patent,

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Respectfully submitted,
BAKER BOTTS L.L.P.

December 16, 2022
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