Jan. 30, 1968 I. S. PASTERNAK ET AL 3,366,419

PROCESS FOR SOLUTION MINING KCL DEPOSITS

Filed Nov. 12, 1964 4 Sheets-Sheet 1

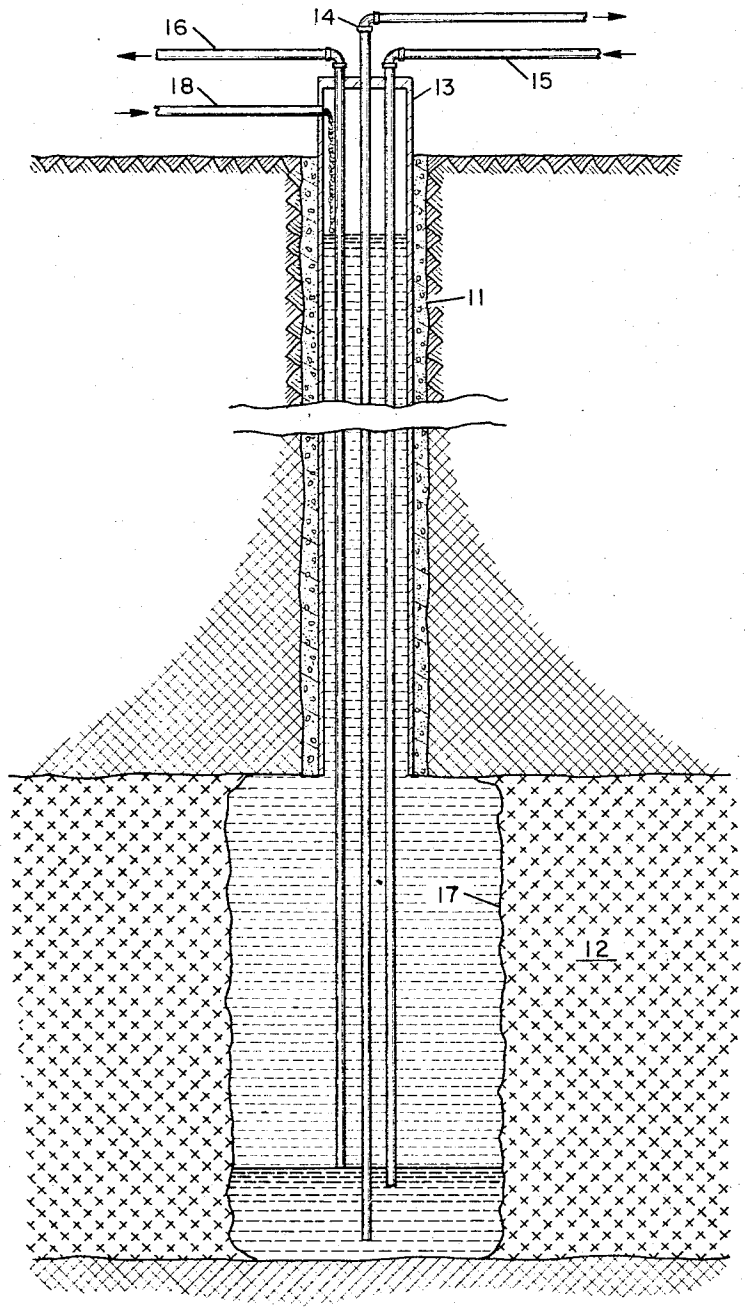

FIG. 1

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Filed Nov. 12, 1964 4 Sheets-Sheet 2

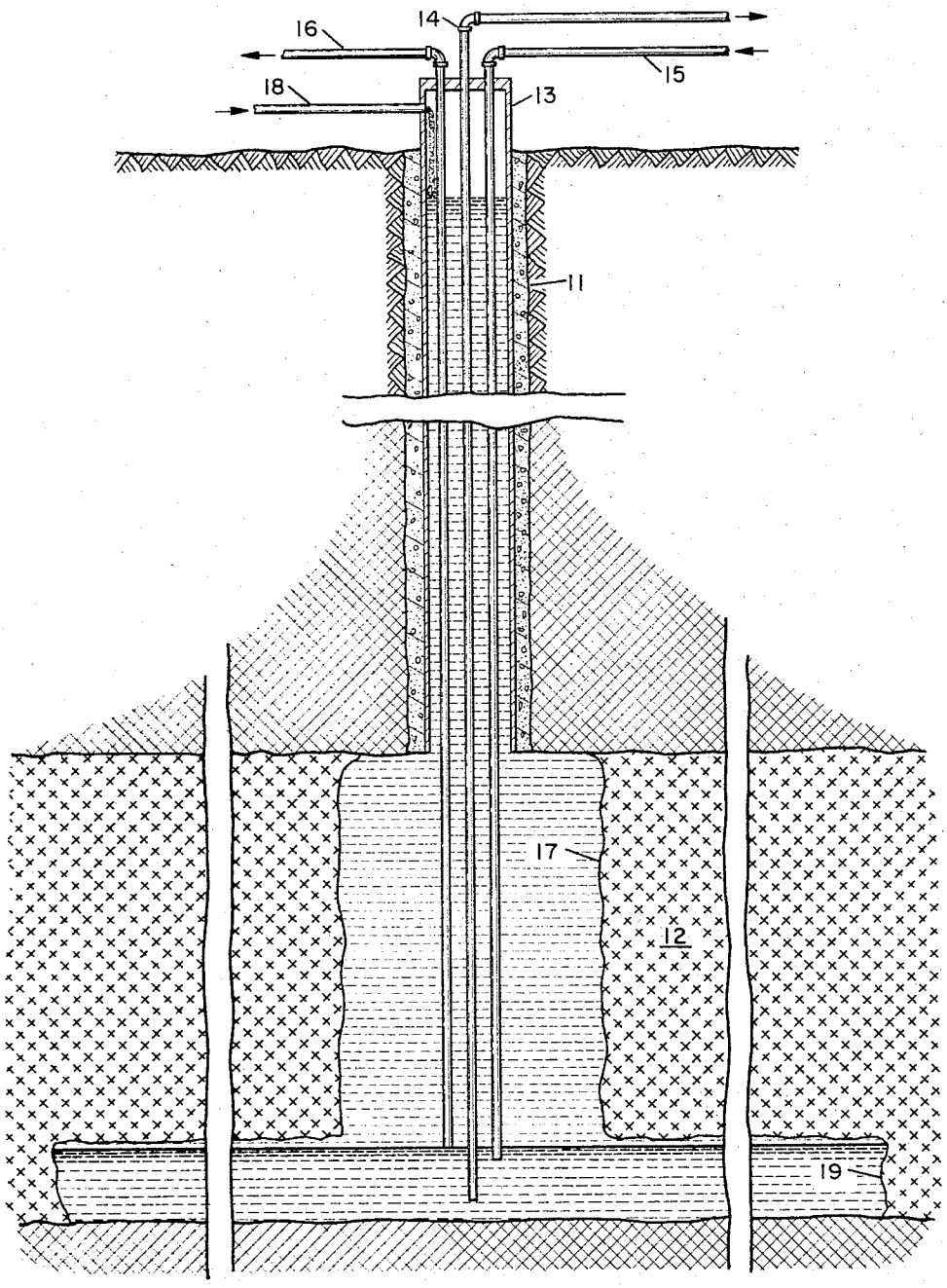

FIG. 2

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PROCESS FOR SOLUTION MINING KCL DEPOSITS

Filed Nov. 12, 1964 4 Sheets-Sheet 3

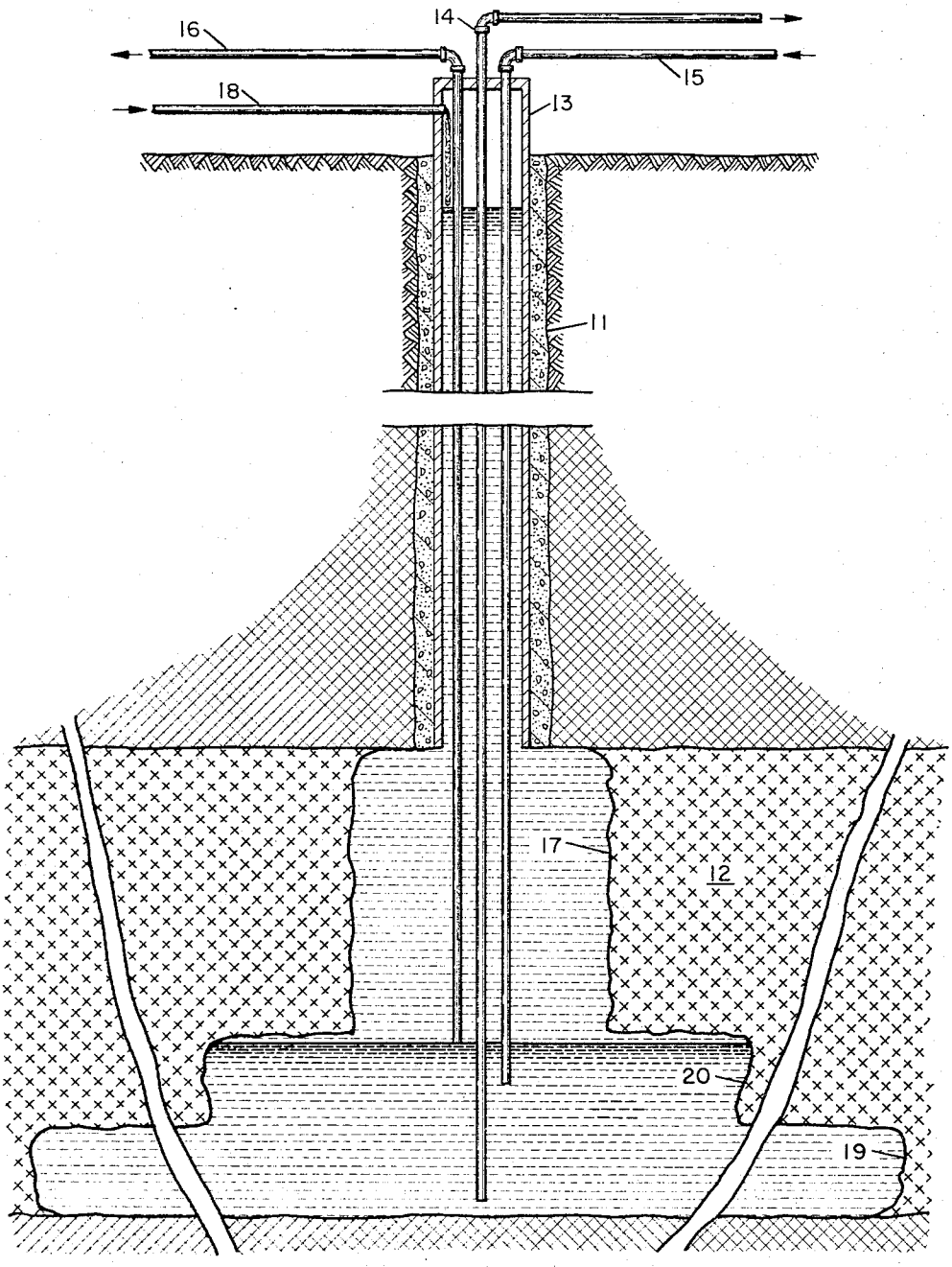

FIG. 3

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PROCESS FOR SOLUTION MINING KCL DEPOSITS

Filed Nov. 12, 1964 4 Sheets-Sheet 4

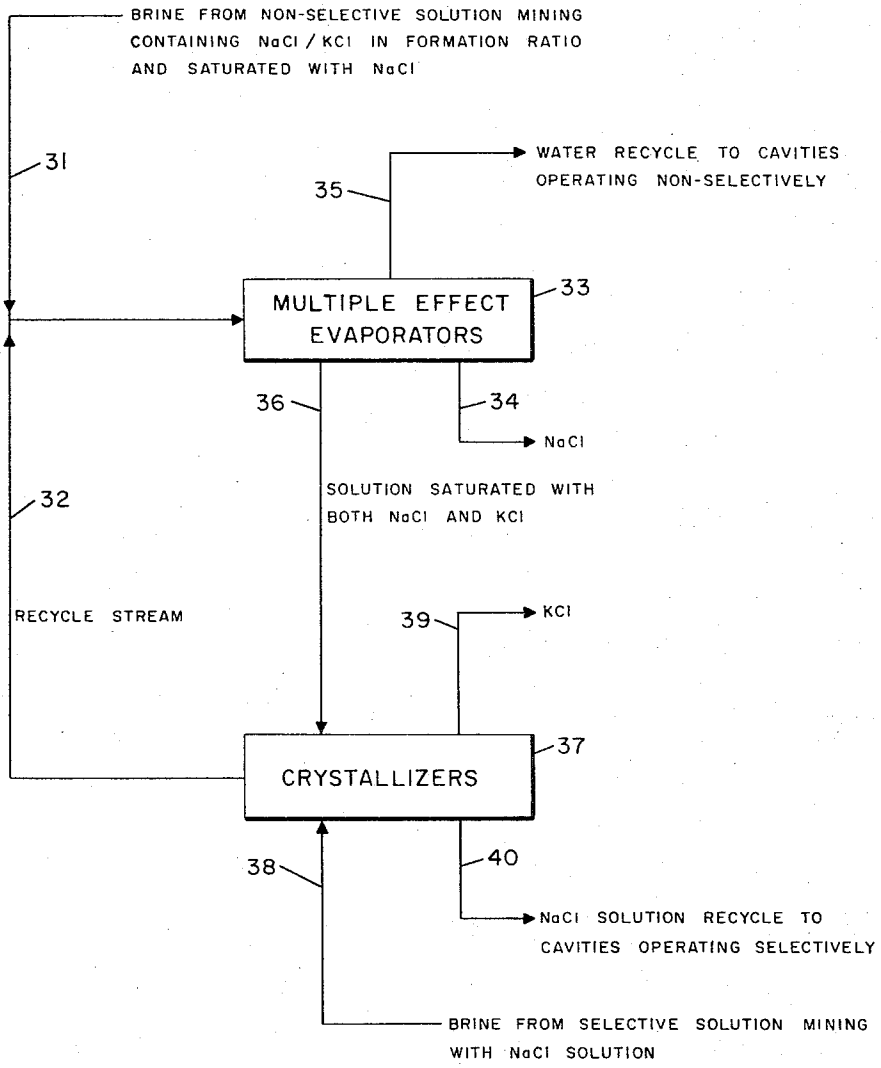

FIG. 4

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United States Patent Office 3,366,419 Patented Jan. 30, 1968

3,366,419

PROCESS FOR SOLUTION MINING KCl DEPOSITS

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12 Claims. (Cl. 299—5)

ABSTRACT OF THE DISCLOSURE

This disclosure relates to methods of solution mining of soluble salt deposits with a suitable solvent and is characterized by a particular method of well formation and by alternating cycles of selective and nonselective dissolution of the salt.

This invention relates to the mining of soluble minerals from underground strata and, particularly, to the solution mining of a soluble salt. More particularly, the invention is concerned with the recovery of potassium chloride from sylvinite by a particular method of solution mining. Most particularly, the invention is concerned with the recovery of potassium chloride from sylvinite by a method of solution mining characterized by a particular method of well formation and by alternating cycles of selective and nonselective dissolution of the salt.

Sylvinite is a physical mixture of sylvite (KCl) and halite (NaCl) which occurs in natural deposits in various places throughout the world. The ratio of KCl to NaCl in the sylvinite deposits varies widely. Not only does the ratio of sylvite to halite vary from one geographic location to another, but the ratio of the two salts may vary at different depths in the same deposit.

A sylvinite deposit sufficiently rich in KCl can be selectively solution mined with a saturated sodium chloride brine. The dissolved potassium chloride may be recovered at the surface simply by cooling the saturated brine, collecting the precipitated potassium chloride crystals and recycling the supernatant brine, with or without the addition of makeup water. However, in the case of KCl-lean ores, once the surface crystals of potassium chloride are dissolved, no further potassium chloride is exposed since the sodium chloride crystals do not drop away from the solution face. Thus, the lean deposits require nonselective mining with water, or with unsaturated brine, whereby the salt formation is dissolved completely producing solutions which are usually saturated with sodium chloride at cavity temperature and which contain potassium and sodium chlorides dissolved in the naturally occurring ratio of the formation.

Heretofore in the art, various methods have been used to mine sylvinite. The usual method for mining sylvinite in order to obtain KCl was to drill from the surface down into the bed of the sylvinite and then inject water into the drilled hole, dissolving the sylvinite, returning it to the surface, and separating the KCl from the NaCl. This procedure required the use of extensive separating means at the surface and added to the cost of the KCl. Another method is that set forth in U.S. Patent No. 3,096,969 for the mining of sylvinite ore which contains KCl in a concentration of at least 15 wt. percent. This method comprised drilling from the surface into the bed of the sylvinite down to a layer which was rich in NaCl, utilizing controlled mining in order to remove the sylvinite laterally from the bottom of the drilled well and

then mining the KCl-rich layer containing more than 15 wt. percent of KCl by filling the lateral excavation with water (or an aqueous solution dilute in NaCl and KCl) and then mining the KCl-rich layer by gradually allowing the water to rise vertically into the KCl-rich layer.

It is the object of the present invention to provide a process of solution mining which comprises a particular method of mining a soluble salt.

It is the object of the present invention to provide an improved method of roof control of the cavity in solution mining by forming a blanket of oil above the solution and by correlating the blanket thickness with the diameter of the cavity as it is being formed.

It is an object of the present invention to provide a process of solution mining which is especially suited for the exploitation of a sylvinite deposit wherein the ratio of sylvite to halite varies considerably. It is a further object of the invention to provide a process of solution mining for the efficient recovery of potassium chloride from sylvinite deposits wherein the selective dissolution of potassium chloride is only partially feasible.

A great many sylvinite deposits are not rich enough in KCl to be selectively mined completely. However, some of these deposits contain rich bands of KCl which can be mined selectively. The present invention is based in part upon the discovery that, in general, sylvinite ores containing less than about 35% sylvite cannot be selectively mined since the removal of potassium chloride does not cause the remaining sodium chloride crystals to drop away from the solution face, whereas deposits containing about 35% or more of KCl can be selectively solution mined because the remaining sodium chloride crystals drop away freely from the solution face, thereby continuously exposing additional potassium chloride. In certain deposits of sylvinite ore, the critical concentration of KCl required for selective mining will deviate from between about 30 to 40%. For example, ores which contain more than the usual amount of insoluble impurities are more amenable to selective mining and may require a KCl content of only 30 to 35%, whereas certain other deposits containing less impurities are not as amenable to selective mining and may require a KCl content of 36 to 40%. The exact percentage required for the selective mining of a given ore is readily determinable, as will appear from later discussion. The usual percentage of KCl in sylvinite which affords the selective mining of KCl in accordance with the instant invention is about 35% based on the total amount of KCl and NaCl in the sylvinite. However, the selective mining of this invention can be carried out when the weight percent of KCl is as low as 30%.

In accordance with one embodiment of the present invention, nonselective mining is carried out by the continuous injection of water during the contact of sylvinite ore containing less than about 30 to 40% KCl, followed by periods of selective solvation of an ore containing KCl in concentrations of 35% or more carried out by the injection of sodium chloride brine. During the latter cycles, crystals of potassium chloride will be selectively dissolved from the ore face leaving the sodium chloride crystals to fall to the bottom of the cavity together with some displaced sodium chloride originally present in the injected brine. When the rich ore band containing more than 30 to 40% KCl has been removed, as determined by analysis of brine sampled from near the top of the cavity, nonselective solution minning using water injec-

tion is again resumed until another rich KCl band is encountered.

In accordance with a further embodiment of the invention, nonselective mining is also carried out initially by the continuous injection of water during the contact of sylvinite ore containing less than about 30 to 40% KCl. When analysis of the sampled brine indicates that a stratum containing at least about 30% KCl by weight has been encountered, the water injection is converted from continuous to semicontinuous flow. Between the periods of water injection, the cavity is shut in to allow successive batches of water to become saturated with both sodium and potassium chloride.

Each batch of water first becomes saturated with sodium chloride and also dissolves an amount of potassium chloride corresponding to the naturally occurring ratio of the formation. Then, because of the high concentration of potassium chloride in the ore, crystals of potassium chloride are selectively dissolved. When this occurs sodium chloride crystals drop away from the face of the ore formation and fall to the bottom of the cavity. At the same time, some sodium chloride will be displaced from solution by potassium chloride and also fall to the bottom of the cavity. The KCl content of the produced solution is thus increased. The sodium chloride crystals falling to the bottom of the cavity remain in a layer of saturated solution throughout the life of the cavity and are not redissolved.

A more complete understanding of the invention may be obtained from the accompanying drawings.

FIGURE 1 illustrates the preferred well completion assembly for practicing the method of the invention. It also shows the conditions which exist in the salt deposit after the initial stages of the process are carried out.

FIGURE 2 illustrates the geometry of the cavity created during the intermediate stages of the process.

FIGURE 3 illustrates the cavity profiles characteristic of latter stages of the process.

FIGURE 4 is a schematic diagram of a surface processing system for use in connection with alternating cycles of nonselective mining with water and selective mining with sodium chloride brine. It is also useful in connection with intermittent water injection.

Referring now to FIGURE 1, borehole **11** is drilled from the surface of the earth through the various layers of the salt deposit to the bottom of zone **12**, the lowermost KCl-containing stratum to be mined. Casing **13** is set in the hole to the uppermost level of the salt deposit. Three tubing strings are then lowered into the casing.

The lower end of tubing **14** is for brine removal and is inserted to a level near the lower boundary of the potash containing ore. The lower end of tubing **15** is for water injection and is positioned about 5 to 10 feet above the brine removal tubing. Tubing **16** is for blanket level control (gas or oil) and is inserted to a level about 1 foot higher than the level of water injection tubing **15**.

Cylindrical chimney **17**, for example, about 10 to 20 feet in diameter is then washed out between the bottom and the top of the potash formation by simply passing water down tubing **15** at a rate of about 500 to 5,000 barrels per day and removing the produced brine by way of tubing **14**. When the top of chimney **17** reaches the uppermost level of the potash-containing formation, as indicated by conventional logging techniques, as well as by a reduced rate of KCl production, the chimney is filled with oil or gas, which is injected into the casing by way of tubing **18** and is maintained at a level about one foot above the end of the water injection tubing.

The development of chimney **17** during the initial stages of the operation allows a relatively large vertical area for dissolution of the potash at the beginning of the undercutting stage of the overall operation. It also serves as a reservoir for the blanket control fluid (oil or gas) supplied from the surface, ensuring an adequate oil supply at a desired level during the subsequent mining stages of the operation. Thus, during the latter stages of the operation, little or no blanket fluid need be supplied from the surface.

With the blanket fluid in place, the washing out of an undercut is begun as shown in FIGURE 2. This stage involves the continued injection of water, and the removal of the resulting potash solution from the cavity while maintaining the level of the oil blanket constant by the injection of oil around the casing and the removal of excess oil from the blanket level control tubing. In many cases, the use of the blanket level control tubing may be omitted and oil added at a rate sufficient to maintain an oil blanket thickness of up to two inches during undercutting. However, such a method of controlling the oil blanket thickness has certain disadvantages as the diameter of the undercut is increased. According to the present invention, the blanket level control tubing **16** is lowered at least about one inch and preferably from 1 to 2 inches for every 50 feet of increasing cavity diameter during the washing out of the undercut. Concurrently with each lowering of the blanket level control tube, an additional amount of blanket fluid is introduced through the casing sufficient to insure an underflow of blanket fluid into level control tubing **16**. Solution of the potassium chloride occurs at the circumference of the cavity below the oil blanket and above the brine outlet tube. This causes undercut **19** to grow in a horizontal direction, its shape being approximately that of a circular disc 5 to 15 feet in height, depending upon the distance between the oil blanket and bottom of the brine removal tube **14**. The undercutting is carried on until the diameter becomes greater than 300 feet at which time the oil blanket is raised about 5 to 15 feet by lifting the oil control tubing. In normal operation, the preferred height of the circular disc and also the distance the oil control tubing is raised is about 10 feet. However, if the particular layer of sylvinite, that is, either the KCl or the NaCl-rich layer, is either more or less than 10 feet thick, then the oil control tubing is moved a corresponding distance thereby providing for cutting substantially within the KCl or NaCl layer.

FIGURE 2 shows a sketch of the cavity configuration at the end of the undercutting period. In order to maintain an undercut of fairly uniform depth, it is important that a high flow rate (at least 4,000 to 6,000 barrels per day, preferably 5,000 barrels per day) be used to prevent the produced brine from becoming saturated. If this is not done, an almost saturated solution will be produced causing the floor of the undercut to rise as the diameter is increased.

After the undercutting stage of the operation, the protective blanket is raised, about 10 feet for example, thereby exposing an extremely large horizontal area for dissolution of the potassium chloride.

FIGURE 3 illustrates successive changes in the cross-section of the cavity during the mining of layer **20** and of subsequent stages of mining in accordance with the alternating cycles of the invention. To prevent dilution of the exit brine with water injected at high rates, the water injection tube **15** is raised such that its outlet end is just below the oil blanket. The separation between the bottom of the injection tubing and the bottom of the brine or water removal tubing is thereby kept at a maximum. The oil level is raised in successive stages of about 10 feet, for example, during which the cycling of nonselective and selective solution mining is continued until a cavity having a height corresponding to the entire salt formation is obtained having a diameter of several hundred feet.

Here it should be noted that periodic samples of brine should be obtained from near the top of a cavity and analyzed for NaCl and KCl in order to determine when to change from a nonselective to a selective period of solution mining or vice versa. This can readily be accomplished by flowing a small quantity of brine up through the oil level control tube in between periods of oil injection into the cavity. This procedure is necessary to

eliminate the long residence time of the produced brine in a large diameter cavity.

FIGURE 4 is a schematic diagram of the surface processing operation used in connection with the alternating nonselective and selective cycles described above. Stream **31** containing brine from a nonselective cycle of the mining process is passed together with recycle stream **32** into a battery of conventional multiple effect evaporators **33**. Feed stream **31** contains produced brine in which sodium and potassium chloride are dissolved in the naturally occurring formation ratio. The brine is saturated with respect to the sodium chloride. In the multiple effect evaporators **33**, sodium chloride crystals are selectively precipitated from the brine and removed at **34**. During this process, evaporated water is recovered and recycled through line **35** to the salt cavity on a continuous or semicontinuous basis, depending upon the stage of the process being carried out as described above.

A brine solution saturated with respect to both potassium and sodium chlorides is passed from zone **33** through line **36** to conventional crystallization zone **37**. The produced brine from a cycle of selective mining is passed by way of line **38** to crystallizers **37** along with the saturated brine from evaporators **33**. The brine of line **38** is that produced from either of the above-described embodiments of the invention. That is, it may be produced from a cycle of continuous sodium chloride brine injection, or it may be produced after a shut-in period of semicontinuous water injection. In crystallization zone **37**, the combined brines are cooled whereby potassium chloride is selectively precipitated and removed through line **39**. The resulting solution is recycled in part through line **32** to evaporators **33**. The remainder of the recycle stream is returned by way of line **40** to one or more cavities being operated on a cycle of selective mining by injection of a saturated sodium chloride brine.

As described earlier, the brine samples obtained periodically from oil level control tubing are analyzed in order to detect the exposure of a KCl-rich stratum in the cavity or cavities being nonselectively mined. Since these brine samples contain sodium and potassium chloride in the naturally occurring formation ratio, the analysis ratio NaCl/KCl gives a direct indication of ore quality currently being mined. Thus, in general, when the analysis ratio falls to about 2.4, preferably 1.9, or below, the nonselective cycle is interrupted and a selective cycle is begun. Conversely, a selective cycle is interrupted when the analysis shows that the solution is becoming unsaturated with respect to KCl, and nonselective mining is resumed.

A critical ratio of 2.4 to 1.9 will not always be satisfactory in the mining of certain ores. As mentioned earlier, some deposits are more amenable to selective mining than others. Thus, at the beginning of a selective cycle, if the KCl content of the sampled brine fails to approach saturation, nonselective mining is resumed until the NaCl/KCl ratio of the ore falls to 1.8, for example, at which time selective mining is again attempted. If the mining solution is not saturated at this NaCl/KCl ratio, nonselective mining is again resumed until selective mining can be conducted. In this manner, a satisfactory ratio of from 2.4 to 1.6 is readily determined for a given ore.

With the beginning of each new stage of the operation, the blanket level control tubing **16** is raised about 10 feet, for example, as mentioned above. After the lower level of the blanket control fluid is raised for the purpose of beginning a new stage of the process, improved roof control is obtained, as before, by the step of lowering the end of blanket level control tubing **16** at least about 1 inch for every 50 feet of increasing blanket diameter during the washing out of layer **20** and each successive layer of the salt deposit. Concurrently with each lowering of the blanket level control tube, an additional amount of blanket fluid is introduced through the casing, sufficient to ensure a continuous underflow of blanket fluid into level control tubing **16**. In this way a continuous driving force is obtained to promote oil (or gas) movement in a horizontal plane, thereby reducing the danger of vertical solvent breakthrough into the cavity roof.

While the present method of mining potassium chloride has been described with particular reference to a single-well operation, it will be readily appreciated that a multiplicity of boreholes or wells may also be utilized. For example, the process may be carried out by sinking two boreholes to the base of the salt deposit, establishing communication between the holes, injecting unsaturated solution through one well and withdrawing saturated solution from the other well.

In connection with such a two-hole system of operation, serious difficulties sometimes arise. The removal of saturated brine through the production tubing string frequently results in a substantial cooling of the brine. Crystallized potassium chloride is thereby caused to precipitate in sufficient quantities to cause plugging of the production tubing.

In accordance with a further embodiment of the invention, this problem is solved by installing a second tubing string in the same hole with the brine production tubing and passing heated water or steam therethrough at a rate which is just sufficient to keep the produced brine at or above cavity temperature, thus preventing crystallization. The bulk of the solvent water is, of course, still injected into the cavity through the other hole. During the mining operation, the water injected through both the holes will dissolve potassium chloride ore, become saturated at cavity temperature and be removed through the brine production tubing. The stream used to heat the produced brine must be injected into the cavity at a level which is high enough to prevent the production of an unsaturated brine by means of dilution in the vicinity of the production borehole.

There are two primary advantages for circulating hot water or steam in the production borehole as above described. While it prevents the crystallization of potassium chloride in the production tubing, it nevertheless allows a large diameter tubing to be used for brine production, because only a small diameter line is necessary for the introduction of a sufficient quantity of hot water or steam to maintain the necessary temperature for the prevention of crystal formation. This causes little or no increase in the horsepower requirements for pumping the total quantity of water into the cavity. Since the brine produced from the cavity during nonselective solutioning must be heated before it is processed in the surface plant, the heat added to the water to keep the produced brine hot may be considered useful as a preheat step for surface processing. Thus, the additional cost of employing the hot water or steam injection is small and the heat exchange required in surface processing may accordingly be reduced in size.

During the nonselective mining period of the process, large quantities of sodium chloride are produced, for which no adequate market usually exists. In accordance with a further embodiment of the invention, the surplus sodium chloride is returned to the cavity during periods of selective solution mining. A return of sodium chloride to the cavity amounts to more than a mere disposal or surplus by-product. Sodium chloride deposited in the solution cavity displaces a brine rich in potassium chloride which otherwise would remain trapped in the abandoned cavity. Moreover, the filling of the cavity with solid sodium chloride provides a significant measure of support for the cavity roof which contributes to the prevention of serious subsidence at the surface.

To obtain a better distribution of suspended sodium chloride throughout the cavity, it is frequently desirable to provide brine slurry injection and brine withdrawal through separate wells laterally spaced some distance

apart. This situation would normally exist where a solution cavity has been formed by circulation through two wells. However, optimum distribution of the surplus salt in the cavity would be obtained when injection and withdrawal wells for sodium chloride disposal are drilled at the extremities of an abandoned cavity.

In the case of reinjecting surplus sodium chloride during periods of selective solution mining, the injected brine slurry is saturated with respect to sodium chloride only and would, therefore, have a density somewhat lower than a produced brine saturated with both potassium and sodium chlorides. Because of this difference in density, conductive forces would tend to promote a good distribution of settled sodium chloride over substantially the entire cavity floor. In this case, brine withdrawal must be from a level near the bottom of the cavity in order to insure saturation with respect to both chlorides.

What is claimed is:

1. A method of recovering a soluble salt from an underground deposit which comprises the steps of:

(a) drilling a borehole from the surface of the earth to the lowest boundary level of said deposit,

(b) establishing fluid communication between the surface of the earth and at least four separate levels within said deposit through said borehole,

(c) injecting a solvent for said salt at the second lowest level of fluid communication and removing the produced salt solution at the lowermost level of fluid communication,

(d) establishing a blanket reservoir of solvent-immiscible fluid from the uppermost level of fluid communication to the next lower level of fluid communication,

(e) continuing the injection of solvent at the second lowest level of fluid communication within said deposit,

(f) raising the lower level of said blanket reservoir in stages thereby recovering salt from the next higher level of the deposit,

(g) continuing the removal of produced solution from the lowermost level of fluid communication, and

(h) increasing the blanket thickness as a cavity is formed by dissolving the deposit laterally.

2. A method as in claim **1** wherein said blanket fluid is a hydrocarbon oil.

3. A method as in claim **1** wherein the blanket reservoir is raised in successive stages of about ten feet.

4. A method as in claim **1** wherein the blanket thickness is increased at least about one inch for every fifty feet of increased cavity diameter.

5. A method as defined by claim **1** wherein the solvent for said salt is a saturated sodium chloride solution when said salt is sylvinite containing at least 30% KCl.

6. A method as in claim **5** wherein the sodium chloride from the sylvinite deposit is returned to the cavity.

7. A method of recovering potassium chloride from a subterranean water-soluble salt deposit containing KCl-rich layers and KCl-lean layers, which comprises the steps of drilling a borehole from the surface of the earth through said salt deposit to the bottom of a selected KCl-containing stratum, establishing fluid communication between the surface of the earth and at least four separate levels within said salt deposit through said borehole, injecting water at the second lowest level of fluid communication and removing produced brine at the lowermost level of fluid communication, thereby forming a generally cylindrical cavity within said deposit, establishing a reservoir of a water-immiscible blanket fluid from the uppermost level of fluid communication to the next lower level of fluid communication, thereafter continuing the injection of water at the second lowest level of fluid communication with said deposit, thereby establishing a disc-shaped undercut at the base of said salt deposit, thereafter raising the lower level of said blanket reservoir in successive stages while continuing the injection of water at said second lowest level of fluid communication within said salt deposit, continuing the removal of produced brine from the lowermost level of fluid communication established within said well bore, and alternating the injection of water within the injection of a saturated sodium chloride brine whenever the analysis of the brine sampled from near the top of the cavity indicates that a band of potassium chloride-rich ore has been encountered containing at least about 35% KCl.

8. In the solution mining of potassium chloride from subterranean water-soluble salt deposits comprising layers which contain more than 35% KCl, alternating with layers which contain less than 35% KCl, the improvement which comprises the steps of nonselectively mining those layers which contain less than 35% KCl by the injection of an aqueous solvent, analyzing the brine samples obtained from near the top of the cavity and, when said analyses indicates the exposure of a layer within said deposit containing more than about 35% KCl, converting from nonselective mining to selective mining of potassium chloride by injecting a saturated sodium chloride brine containing surplus sodium chloride slurried therewith.

9. In the solution mining of potassium chloride from subterranean salt deposits comprising layers which contain more than 35% KCl, alternating with layers which contain less than 35% KCl, the improvement which comprises the steps of injecting a saturated sodium chloride brine in contact with the potassium chloride-rich layers, said sodium chloride brine containing surplus sodium chloride slurried therewith, and alternately injecting unsaturated sodium chloride brine or water in contact with layers containing less than about 35% potassium chloride.

10. In the solution mining of potassium chloride from subterranean salt deposits comprising layers which contain more than 35% KCl, alternating with layers which contain less than 35% KCl, wherein communication with said salt deposit is established through an input well and an output well laterally spaced therefrom, the improvement which comprises injecting a slurry of surplus sodium chloride contained in a saturated aqueous solution of sodium chloride through said input well in contact with the potassium chloride-rich layers alternately, injecting unsaturated sodium chloride brine or water into contact with layers containing less than about 35% potassium chloride, removing a resultant brine through said output well, and passing a small amount of hot water or steam in indirect heat exchange with the stream of produced brine as it is being removed through said output well bore.

11. In the solution mining of potassium chloride from subterranean salt deposits comprising sodium chloride and potassium chloride and comprising layers which contain more than 35% KCl, alternating with layers which contain less than 35% KCl, the improvement which comprises the steps of nonselectively mining those layers which contain less than 35% KCl by the continuous injection of an aqueous solvent, analyzing the brine samples obtained near the top of the cavity and, when said analyses indicate the exposure of layers within said deposit containing more than about 35% KCl, converting from nonselective mining to selective mining of potassium chloride by using an intermittant flow of the aqueous solvent, whereby the time between each successive flow of solvent is sufficient to enable said solvent to become saturated with sodium chloride and potassium chloride.

12. In the solution mining of potassium chloride from subterranean salt deposits comprising sodium chloride and potassium chloride and comprising layers which contain more than 35% KCl, alternating with layers which contain less than 35% KCl, the improvement which comprises the steps of non-selectively mining those layers that contain less than 35% KCl by the continuous injection of an aqueous solvent, analyzing the brine samples ob-

tained near the top of the cavity and, when said analysis indicates NaCl/KCl ratio of at least 2.4, converting from non-selective mining to selective mining of potassium chloride by using an intermittent flow of the aqueous solvent, whereby the time between each successive flow of solvent is sufficient to enable said solvent to become saturated with sodium chloride and potassium chloride.

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