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THE AMERICAN POTASH INDUSTRY AND ITS PROBLEMS[1]

By John E. Teeple

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Economic conditions growing out of the war left two chemical foundlings on our shores. One was the dye industry and the other the potash industry. The first proved to be a noisy one, and we have been kept rather well informed of its progress, its possibilities, its hopes, and particularly its needs. The potash industry has been far less noisy, although we have been kept informed from time to time that we have such an industry. But when we ask, "How much of a potash industry have we?" —"Is it a permanent one?" and "Can it either now or in the future compete with the German potash industry, or must it temporarily or always receive protection as an infant industry in order to exist?" we find a considerable divergence of opinion.

I want to discuss some things regarding the progress and problems of this American potash industry, its present position, and its hopes.

POTASH IN THE UNITED STATES

This is primarily an agricultural country. It normally consumes 250,000 tons K_2O per year, which is equivalent to 400,000 tons of 100 per cent KCl (potassium chloride). This figure, of course, includes all grades of potassium salts brought into this country, and over 90 per cent of the total is used on the land as fertilizer. Before the war we produced no potassium salts at all in this country, with the exception of an infinitesimal amount of potash leached from wood ashes. Even the caustic potash made in this country was made from imported potassium chloride. When the war shut off commerce with Germany and the country awoke to the fact that it had no potash and that it must have it to produce the large crops that were needed, our Government sent out urgent requests to hasten the discovery and the production of potash from every possible source. These urgent requests, together with the high price which could be obtained for any salts containing potassium, resulted in the installation of plants to work the natural brines of Nebraska, California, and Utah; the dust from cement kilns and blast furnaces; the waste liquors from distilleries and beet-sugar factories; the alunite deposits of Utah; the leucite deposits of Wyoming; the kelp fields of the Pacific coast; the wood ashes of Michigan; and even the greensand of New Jersey. In all we find a total of over 100 different plants built to produce potash from these sources. In 1918, the banner year, 123 different plants operated, giving a total production of over 54,000 tons K_2O. In 1919, with the fall of the price of potash, this production dropped to about 30,000 tons; and, while the figures are not yet available for 1920, the production will be probably in the same neighborhood—that is, something like one-eighth of the country's requirement.

Out of the 128 plants reported as producing in 1918, only 43 were reported as producing in 1920. With the price of potash in 1921 still lower than it was in 1920 we may expect a still greater falling off in the number of producing plants, and possibly in the total output.

Up to the present time probably 70 per cent of the total production of potash in this country has come from natural brines. These natural brines include a whole series of lakes in Nebraska, the Salduro Marsh in Utah, and Searles Lake in California. There are eight plants working on the Nebraska brines, one on the Salduro Marsh, and three on Searles Lake. Just now, of the eight Nebraska plants five are closed down and only one of the three plants on Searles Lake is in regular operation.

SEARLES LAKE POTASH

The oldest and largest of the plants working on Searles Lake and also the largest producer of potash in the United States is the American Trona Corporation. In 1918, the year during which the United States produced more potash than it has ever done before or since, this one plant was responsible for about one-seventh of the entire production of the country. To-day this one plant is probably responsible for about 30 per cent of the production of the United States, and as it seems to be making more progress and spending more money in studying its problems than any other potash organization, a study of its difficulties encountered and results achieved should give us some basis for predicting the future. I hope that a study of these difficulties will be interesting from a chemical and engineering point of view, and at the same time it will show us why the industry has not progressed faster than it has, why potash is not yet as cheap as it was before the war, and why we may expect that with moderately good fortune a permanent potash industry can be founded here.

When I first assumed responsibility for the operations of this concern about a year and a half ago, I found a going plant which represented an investment not far from $10,000,000, but which still did not seem particularly well adapted to the purpose for which it was built. It was producing at that time only a moderate tonnage of potassium chloride, averaging less than 70 per cent in purity, and its production costs were high. Many different processes had been tried at this plant in a desultory manner, many millions of dollars had been spent, not always wisely, and even with such a brief history behind it the plant was being run on tradition rather than on information. This is not said in a spirit of petty criticism, for we all realize that pioneer work when done under pressure for production is not usually accompanied by careful, painstaking work, calm judgment, and economical operation. The whole spirit under such conditions is one of snap judgment and lavish expenditure, and a certain amount of both is excusable and unavoidable. In any case, by the middle of 1919 the plant had passed the pioneer stage and the press for production where such modes of operation could be excused, and was entering on a period where an entirely different

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[1] Based on addresses given before the Rochester and Cornell Sections of the American Chemical Society, January 24 and 25, 1921.

method of handling the problems was necessary if the plant was to live. Two essential lines of work seemed to offer themselves for me to undertake at this stage. One was to study the existing plant and put it into a position to produce as much material as possible of the best quality and at the cheapest price consistent with the minimum expenditure of money for plant extensions and changes. The second line of work was to make a complete study of Searles Lake brine so as to know definitely how it should be handled in the existing plant, or in any other plant that might take its place. Both these lines of work have been of intense interest to me, and I hope as I report progress of them to you they will have some measure of interest to you.

THE SEARLES LAKE DEPOSIT

Searles Lake is a bed of crystallized salts about 11 or 12 sq. mi. in area and 60 to 70 ft. in depth. The salts are permeated throughout with brine that has come to equilibrium with the salt deposit. The lake is fed continually by underground streams which enter the salt bed probably from below, come to equilibrium with the salts, and evaporate their excess water at the surface or furnish brine for the potash plants located on the shores of the lake. In summer evaporation keeps the brine an inch or so below the surface of the salt body so that the whole lake becomes a level and luxurious automobile course. In winter when evaporation is slower the brine stands an inch or so above the surface of the salt. The salt crystals consist of:

Halite ($NaCl$)
Mirabilite ($Na_2SO_4.10H_2O$)
Thenardite (Na_2SO_4)
Trona ($Na_2CO_3.NaHCO_3.2H_2O$)
Tinkal ($Na_2B_4O_7.10H_2O$)
Hanksite ($9Na_2SO_4.2Na_2CO_3.KCl$)
Glaserite ($Na_2SO_4.3K_2SO_4$)

This list of salts comprises practically the entire crystal body. Neither KCl nor Na_2CO_3 exists as such in the salt body, nor do calcium or magnesium salts. But below the salt body itself we find insoluble salts of calcium and magnesium where they are apparently precipitated before the inflowing water reaches the salt bed. The brine which permeates the whole salt body is naturally kept in equilibrium with the salts by solution and precipitation. Since the temperature of the brine taken at a point below the surface is fairly uniform at 23° C. summer and winter, it follows that the brine drawn out for use in the plant is of uniform composition.

The lake is probably the finest natural situation to study and plot equilibria that a chemist ever had. The large body of salts and brine serves as an insulator or heat reservoir. The varying evaporation provides a thermostat, and centuries of time have passed since the whole mass reached a static condition. The composition of the brine as it is drawn from the middle of the lake, probably 50 ft. below the surface, is as follows, when expressed in conventional symbols:

	Per cent
$NaCl$	16.54
KCl	4.82
Na_2CO_3	4.17
$NaHCO_3$	0.52
$Na_2B_4O_7$	0.85
$Na_2B_2O_4$	0.85
Na_2SO_4	7.16
H_2O	65.09

This composition does not vary more than a few hundredths of a per cent at any time throughout the year. There are a few minor annoyances, like sodium sulfarsenite, sodium bromide, sodium iodide, organic matter, etc., but they need not be considered now.

The quantity of this brine is appalling. It has been estimated to contain over 12,000,000 tons of potassium chloride, and the salt body itself probably contains more than twice as much more undissolved potassium, computed as potassium chloride.

EQUILIBRIUM DIAGRAMS

Looking back at the brine analyses you will note that figures are given for the content of bicarbonate and metaborate. These figures were arrived at by Mr. W. E. Burke, of our research department, after a complicated calculation based on certain assumptions. We do not guarantee their accuracy, but they are the best approximation we have been able to make. We know that bicarbonate is present because the brine is in equilibrium with trona, and we know metaborate is present because borax in the presence of sodium carbonate is known to form metaborate. Up to the present, however, we have found no direct and reliable method of determining metaborate and bicarbonate in the presence of borax and carbonate.

Looking again at the brine analyses it is obvious that the two most valuable constituents, potassium chloride and borax, are present in relatively small amounts, and hence that we must concentrate them to make them marketable. How shall this be done? We could, of course, evaporate off all the water. This would leave a salt containing nearly 14 per cent KCl and so corresponding to the German carnallite, but as this salt would also contain nearly 5 per cent $Na_2B_4O_7$ it would not be marketable. In the case of Nebraska brines, however, where the borax content is negligible, this method is actually in use, and most of the Nebraska potash that has heretofore come on the market has been subjected to no other operation than simple evaporation, or evaporation and drying. In our case this is impossible. We must concentrate both potash and borax in some manner, and since neither can be precipitated to advantage we must turn our attention to the removal of the undesired salts at the same time that we evaporate the water. To do this we can consider evaporation in solar ponds, in spray ponds, in spray towers, or in vacuum pans, or we can consider refrigeration. We have tried all these methods. At present we are evaporating in triple-effect vacuum pans, because this was the equipment we found in the plant. What we shall do ultimately is still an open question. In this question of concentration we find our equilibrium diagrams of supreme importance. It would be a prolonged task to plot the equilibria, taking into consideration all the brine constituents, so we undertook to plot simply the system of sodium and potassium as chloride, sulfate, and carbonate, at various temperatures between 0° and 100°, and for all possible saturation concentrations. This work is now well under way. So far, including double salts, we have located 7 quadruple points, 14 triple points, 10 double points, and 8 single points. I do not know how many more there are; possibly a combination of a mathematician and a physical chemist could figure it out. This equilibrium work has been done chiefly by Mr. Harold de Ropp, of our research department, and as it has progressed it has proved most useful in enabling us to understand what we were doing, and hence in permitting us to improve intelligently on our practice. It is proving still more valuable in helping us to avoid useless experimentation. When the equilibrium work is completed we should be able to predict just what would happen in any suggested operation or "process." This work will all be published at the proper time and should prove a valuable contribution. It will be noticed that in this equilibrium study we eliminated borax, metaborate, and bicarbonate for the sake of simplicity. Even physical chemists have their limitations. While they handle the fourth dimension with impunity they sometimes hesitate to undertake an equilibrium model requiring the use of a fifth, sixth, or seventh dimension. We are seeing what can be done with these neglected salts in a separate study.

DOUBLE SALTS

The bête noir in concentrating Searles Lake brine and similar brines for potash is the formation of double salts, particularly glaserite or aphthitalite ($Na_2SO_4.3K_2SO_4$). Given a solution containing KCl and NaCl, the separation is fairly easy. Evaporation by boiling precipitates NaCl and enriches the KCl in solution. Cooling then precipitates KCl. Given KCl and Na_2SO_4, however, the picture is entirely different. We may imagine a reaction equation as follows:

$$6KCl + 4Na_2SO_4 + \text{water} \rightleftarrows 3K_2SO_4.Na_2SO_4 + 6NaCl + \text{water}$$

The transition point for glaserite is just about 3° C., so if we evaporate at a temperature above 3° C. the solid phase is glaserite and NaCl. If we evaporate below 3° C. the solid phase is Glauber's salt and KCl. In neither case have we made any real concentration. In order to avoid this precipitation of potassium as glaserite at any temperature we must keep the concentration of Na_2SO_4 below the point where it would be in equilibrium with glaserite at that temperature. This did not seem an easy thing to do, but Mr. Burke discovered the remedial agent in the form of another double salt, $Na_2CO_3.2Na_2SO_4$. This double salt has not previously been described in the literature. We have accordingly called it "burkeite" and it will be described in detail in a separate paper. It forms well-defined crystals of very high luster and definite composition. Its transition point is about 24° C. Below this temperature it does not seem to exist. As the temperature rises its solubility decreases. This, in fact, led to its discovery. In July 1919 my attention was called to a beaker of brine which was being gently warmed far below evaporation temperature, and which was forming beautiful crystals of a salt. Various suggestions were made that these were crystals of sodium chloride, sodium sulfate, sodium carbonate, or mixtures, and, while I suggested that it might be well to do a little analytical work and find out just what they were, it made no particular impression on me or anyone else, because our brines are so thoroughly saturated at all stages that something is always separating out of them, whether we cool them or warm them or evaporate them or merely look at them. Later, Mr. Burke's analyses showed that we had to deal with a true double salt. Now, the discovery of this double salt, together with a study of our equilibrium diagrams, showed us how by proper manipulation of our sodium carbonate concentration we could always keep the concentration at which sodium sulfate was in equilibrium with burkeite below the concentration necessary to form glaserite. In other words, the sodium sulfate present could be made to separate from the solution as burkeite and not as glaserite, so the potash could be kept in solution and concentrated to saturation. It was necessary only to mix the raw brine with other liquors rich in carbonates and evaporate the mixture in triple-effect vacuum pans, keeping the carbonate always at proper concentration,[1] to depress the sulfate below the point of glaserite formation. Under these conditions sodium chloride and burkeite separate continuously until we arrive at a hot concentrated liquor which contains more potassium chloride and borax than it does all other salts combined, and which on cooling to 30° deposits essentially only potassium chloride and borax.

SEPARATION OF POTASSIUM CHLORIDE AND BORAX

Unfortunately, no one wants a mixture of potassium chloride and borax. During the war under government urgings for production, such mixtures were sold more or less regardless of their borax content and were used in fertilizers, and the experts are still arguing whether the borax did or did not harm certain crops. In any case, it seemed more profitable to separate the borax from the potassium chloride and sell it independently for other purposes than for fertilizer. This separation proved a difficulty for some time, but the laziness of borax finally came to our assistance. It was found that by cooling the hot solution rapidly and quietly to about 30° a crop of potassium chloride separated without a crystal of borax in it.[1] The mother liquor from this crop without further cooling but with agitation, or with time, deposited a good crop of borax with very little potassium chloride, and this little was easily removed. It was not simple to design a cooler that would act promptly and effectively, but a new one recently installed according to ideas worked out by Mr. H. S. Emlaw, the general manager of the plant, seems to meet all requirements and furnishes a borax which is uniformly over 99.5 per cent in purity, and a potassium chloride which at present averages over 92 per cent KCl with less than 0.5 per cent borax, and which we expect in the near future to average 96 or 98 per cent KCl with still lower borax content. In fact carloads of potassium chloride shipped now, taken at random, very frequently run 97 or 98 per cent purity. One carload, the analysis of which I noticed recently, ran 99.78 per cent KCl. The impurities present are, of course, chiefly sodium chloride with a little sodium sulfate, sodium carbonate, and borax. In the matter of purity, these products at present leave little to be desired, and their quality is still being improved. When compared with the German potash furnished to this country in previous years they represent an enormous advance in purity.

FOAMING

Now that we have seen our way through the operation of producing potash and borax from these brines, which is after all an amazingly simple one of evaporation and crystallization, let us go back to a few of the other troubles. One very serious trouble has been the foaming of the brines during evaporation. As soon as boiling in the vacuum pans was well under way a voluminous, persistent, pernicious foam, like soapsuds, would rise from the brine and pass over with the vapors to the calandria of the next effect, or to the condenser, causing serious losses of the products we were trying to save, and slowing down the operation materially. A synthetic brine containing all the known inorganic constituents of the natural lake brine did not foam seriously, in fact, it boiled as quietly as a tea kettle, so we inferred that the foam producer was organic. On this assumption we tried oxidation, reduction, chlorination, electrolysis, all the usual remedies, with no satisfactory result. We did find, however, that the foam producer could be entirely removed from the brines by treating them with decolorizing carbon, or bone-black, or in some cases with clays, but all these methods were expensive both in installation and in use. We found further that any slight coating of oil on the surface of the brine, whether the oil was animal, vegetable, or mineral, completely stopped the foaming so long as a film of oil stayed on the surface; and this would have been the natural and complete remedy for foaming had it not been for one unfortunate and unexpected cause of trouble. Any oil that was used attached itself to the burkeite crystals and acted as a flotation agent, keeping the burkeite suspended instead of allowing it to settle, and the oil itself being thus continually removed from the surface had to be as continually replaced. This definitely eliminated the possibility of using oil. Centrifugal foam separators were, of course, installed, and various mechanical devices tried. These were necessary but not sufficient. Various methods of operation were likewise attempted, such as carrying the liquor at a low level in the evaporator tubes. This absolutely prevents foaming, but on the other hand it causes the tubes to salt up rapidly and put the evaporator out of commission.

Things being in this unsatisfactory condition, then, it seemed wise to determine, if possible, what this organic material was and whence it came. To answer the last question a short study of the watershed was made to learn what organic matter might be at hand in this desert region of so potent a quality that 2 to 4 in. of annual rainfall could bring it into the lake in quantities sufficient to cause such a disturbance. The only vegetation occurring in considerable quantity seemed to be the desert sage, the cactus, and the creosote bush. The creosote bush (*Larrea mexicana*) was found to be the offender. The leaves of this bush appear to be heavily varnished, and an extract of the leaves and branches when added to synthetic brine causes

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[1] This is the subject of patent application.

it to foam just as the natural brine does. Investigation of the creosote bush showed that the foam-producing constituents were certain resins composing this varnish, and certain saponins, particularly acid saponin, which were easily extracted from the plant. With this as a basis a careful study was made of the whole question of foams, and quite a large number of materials were found which would stop the foam when added in almost infinitesimal amounts.[1] For example, while ethyl alcohol increases the amount of foam in natural brine, caprylic alcohol decreases it; and if we go to still higher alcohols, like cholesterol, an exceedingly small amount—say 10 p. p. m.—has a very marked effect in stopping the foam. Gum arabic increases the foam, and a colloid gum of unknown origin decreases it. Certain esters, like amyl valerianate, check the foam, as well as do certain organic acids, sulfonic acids, and their salts. This field is still being investigated for different classes of materials, like phenols, amines, etc. As far as we can see at the moment, it is either a surface-tension phenomenon or a precipitation phenomenon by a colloid of opposite sign, or both. In any case, our troubles at the plant from this cause are apparently entirely in the past. A considerable part of the early work on this subject was done by Mr. Frederic Vieweg, the assistant manager, and the later work by Mr. Burke and Mr. Clark M. Dennis of the research department. Credit for suggesting and working out the details of the means now actually in use goes to Mr. Russell W. Mumford, in charge of research and development work at the plant, and this will all be reported on in detail at the proper time.

VACUUM DISTILLATION

One interesting feature of this problem of foam, which was first called to our attention by Mr. A. L. Webre, was the fact that the boiling temperature of the liquor in our vacuum pans did not agree with the boiling temperature of the same brine obtained in the laboratory at the same pressure. The discrepancy was found to be due to the fact that in the plant the foam passing through the vapor lines from the vacuum pans caused so much friction, and hence back pressure, that the vacuum which we read in the vapor lines was not the actual vacuum in the pans, and this difference was sufficient to cause 12° F. rise in the boiling point of the brine. Now that the foaming is stopped this difference no longer exists.

OTHER PROBLEMS

We have mentioned above a few of the problems that have been solved. Anyone familiar with this kind of work will realize at once that the solution of these few problems in a going plant represented a very large amount of work by a considerable organization. We have succeeded in improving the quality of the product to a point where we can satisfy the requirement of almost any buyer no matter what his specifications are. We have succeeded in cutting the cost of production very materially below what it was in previous years, and this has been done even in the face of rising costs of fuel, labor, and supplies. But it will be realized at once that there are many other problems still under investigation whose solution will be necessary before the work can be considered finished. For example, in our evaporators we have never yet succeeded in getting the capacity, *i. e.*, the pounds of water evaporated per square foot of heating surface per hour, anywhere near up to the point where it should be and where we hope to get it. We use iron tubes in the evaporators, and we must in some way increase the coefficient of heat transmission through the iron. Investigation has already shown that we can get this increase by giving a proper circulating motion to the brine, but how to give this proper circulating motion through some 30,000 tubes of our evaporators is an engineering problem still in course of study. We should like to use copper tubes in the evaporators because its coefficient of heat transmission is considerably higher than that of iron, but our brines are alkaline and contain small amounts of ammonia which renders the use of copper impossible on account of corrosion. The brines contain some sulfur which seems to be given off as hydrogen sulfide or volatile sulfides. This attacks the iron on the steam side of the tubes, converting it into iron sulfide and ultimately making replacement necessary. We know how much water a pound of fuel oil or a pound of steam should evaporate from our brines in triple-effect evaporators. We are as yet unable to approach this figure, and even after making due allowance for the heat of solution of the salts that separate, and the specific heat of the salts themselves, there is a very considerable unexplained discrepancy. If we make 100 per cent recovery of all the potassium chloride in the brine it will be seen that we must evaporate about 14 lbs. of water for every pound of potassium chloride produced. Our recoveries are still a very long distance from 100 per cent. We have made very good progress in improving the per cent recovered, but we still have a long way to go. Consequently, at present we still must evaporate a good deal more than 14 lbs. of water to produce 1 lb. of KCl, and, being located as we are, in a place where the only fuel available is rather expensive fuel oil, it becomes obvious that the cost of fuel and the cost of steam production in general is one of our largest items of expense and one of the places where we can still make the largest saving. I consider the problem of chasing the elusive B. t. u. one of the most important problems now facing us, and this work is under way. We have not yet reached a point where our cost of production is as low as 75 cents per unit K_2O, which was about the minimum selling price of German potash before the war, but we are beginning to see where we may get within shooting distance of it. It will probably take two or three years yet to work out this problem to the point where we are producing potash at Searles Lake in large quantities as cheaply as it can be produced there. When this point is reached I do not think we need seriously fear German or any other competition.

TEMPORARY PROTECTION

The two largest items of cost in production we have at the present moment are fuel and freight, and if we can get any kind of ultimate coöperation from the oil producers and from the railroads we shall be able to supply a large part of this country's need of potash without any protection. Do we need protection until this work is finished? It is probable that we do. Germany's need of money and goods is a most serious need. There will be great temptation for her to convert some of her supplies of potash into immediate cash. Probably no potash plant in America could sell its product to-day at $1.00 per unit and cover its cost of production. If this industry can be coddled for about 3 yrs., until investigation and development work are finished, it is probable that we shall have a very considerable production in the United States on a sound basis and at a fairly cheap price. If it is not coddled the chances are that production will almost cease, and our experience shows that where production ceases there is little incentive to keep expending money on research and development work. Prices of potash during the past year have averaged close to $150 per ton of KCl. On the basis of our average consumption this represents close to $60,000,000. Before the war this would have represented around $20,000,000. The size of either figure makes it well worth while to encourage the growth of such an industry in this country, since it apparently has every prospect of being able to live alone and do its own fighting once its development period is over. We have discussed only the problems of one single plant. All the other plants have their own problems which they are working out in their own way, and in very many cases they will no doubt arrive at as favorable a conclusion as we ourselves hope to reach, if they are permitted to have this period of development.

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[1] This is the subject of patent applications.