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Howson and Howson

April 8, 1952

H. J. HEGAN
CONTINUOUS PROCESS FOR THE PRODUCTION
OF ANHYDROUS GLAUBER'S SALT

2,592,139

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FIG. 5.

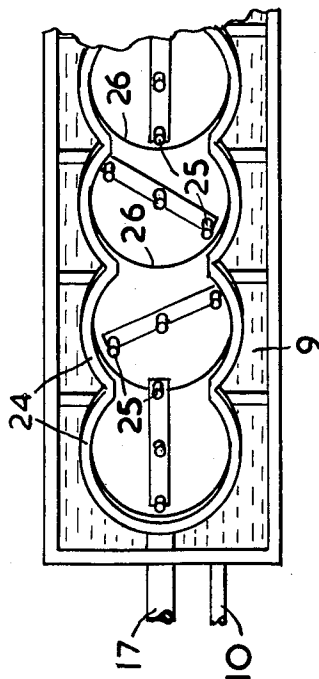
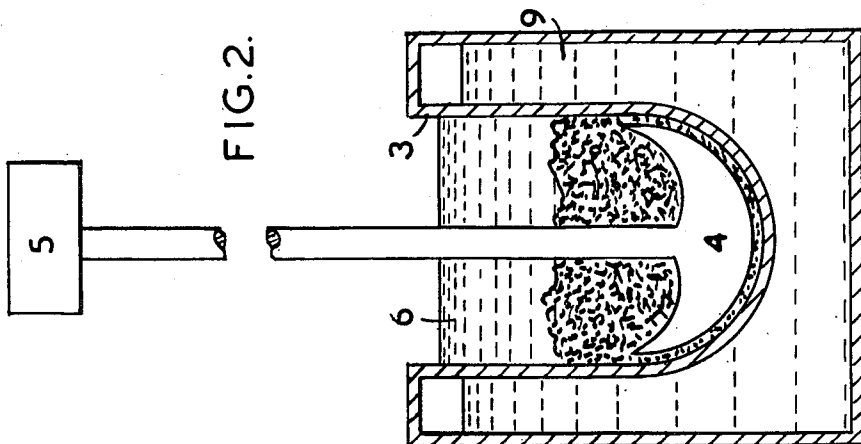


FIG. 2.



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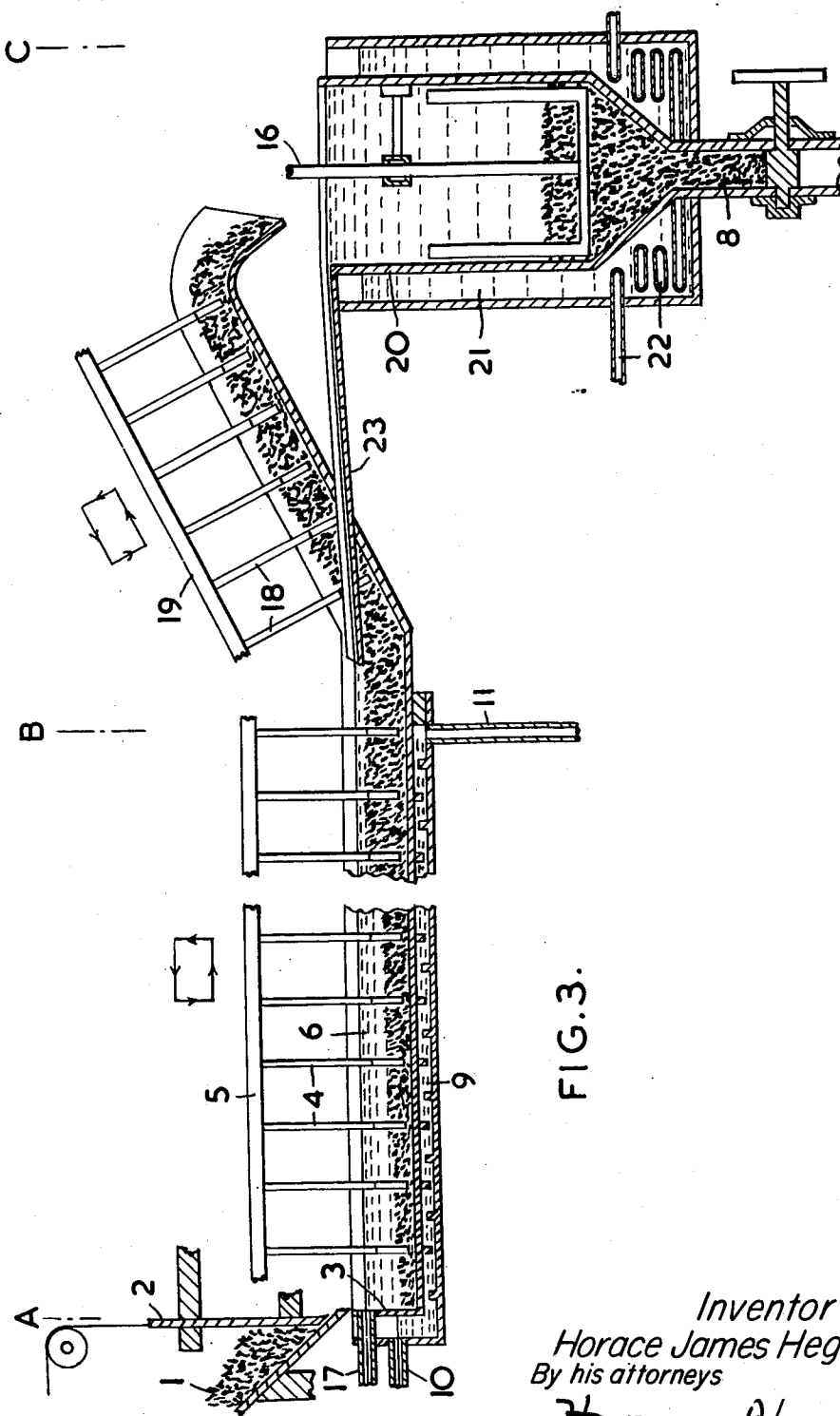


FIG. 3.

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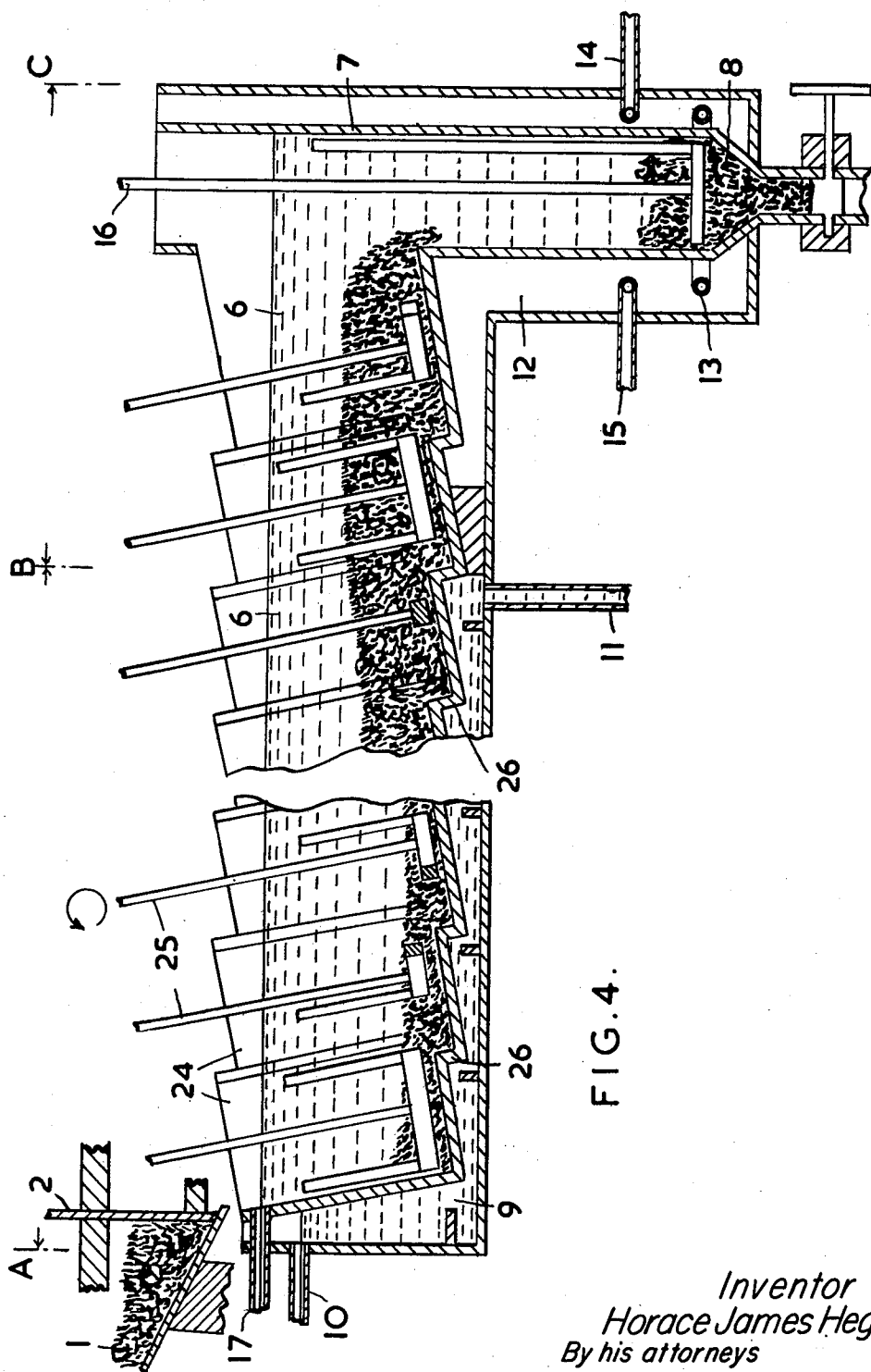
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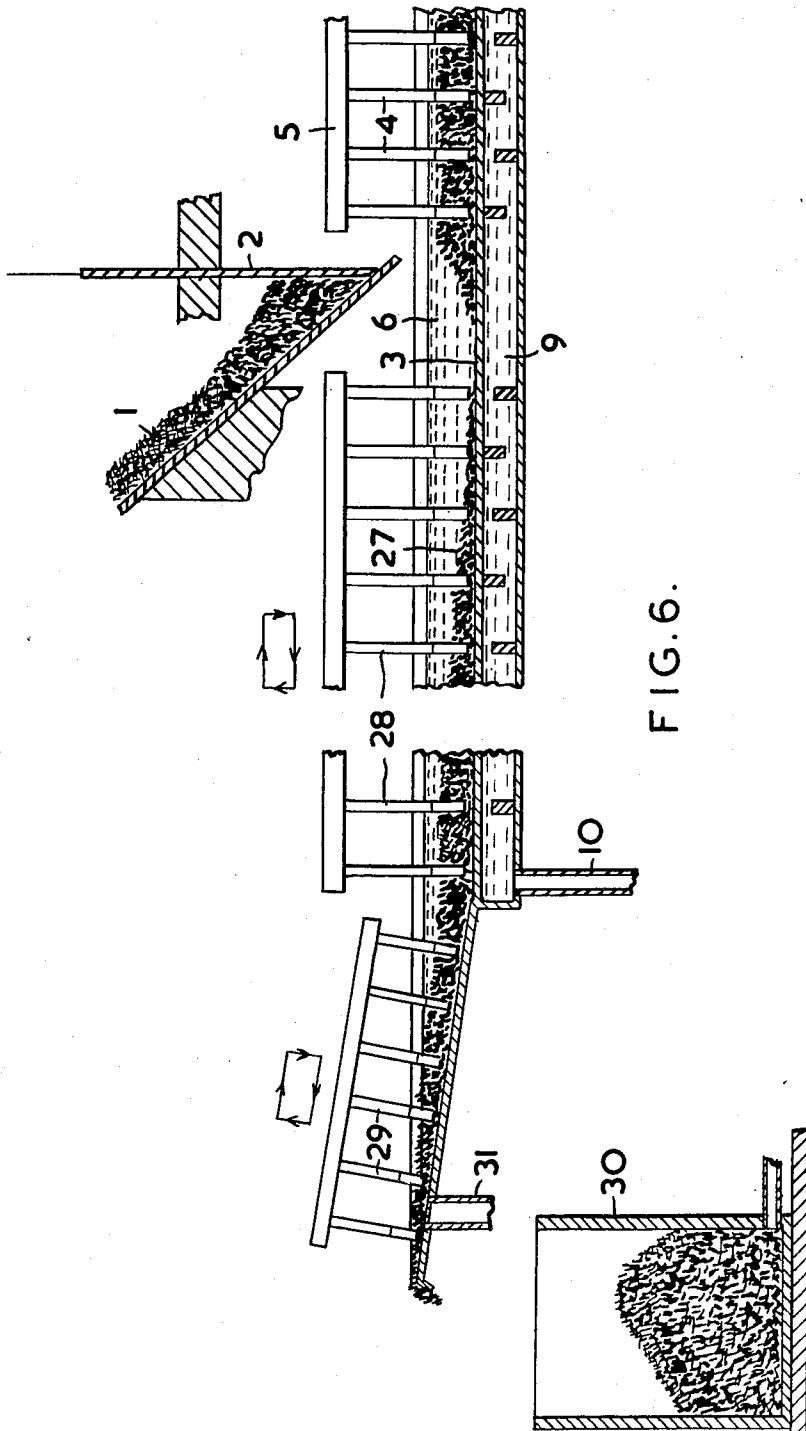
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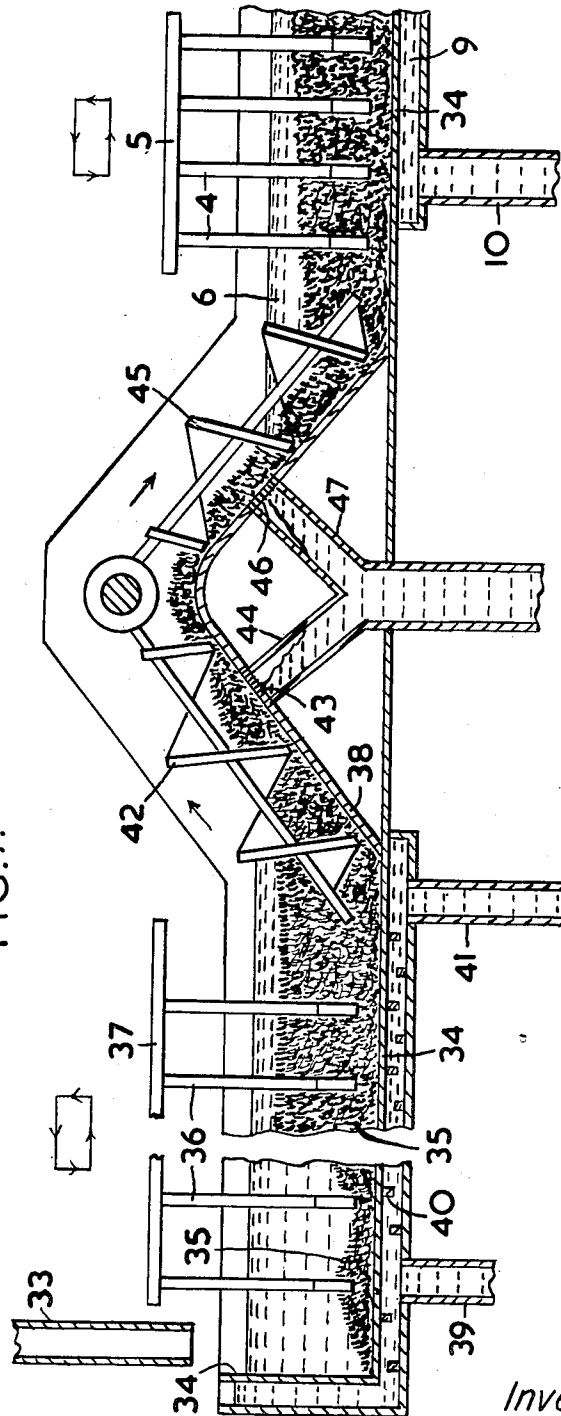
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FIG. 7.



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UNITED STATES PATENT OFFICE

2,592,139

CONTINUOUS PROCESS FOR THE PRODUCTION OF ANHYDROUS GLAUBER'S SALT

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Application June 6, 1949, Serial No. 97,414
In Great Britain July 2, 1948

4 Claims. (Cl. 23—121)

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This invention relates to the conservation, recovery and purification of chemicals from viscose rayon spinning baths.

In the viscose spinning industry it is standard practice to extrude viscose into coagulating baths containing sodium sulphate, sulphuric acid and zinc sulphate; the chemical processes taking place in the bath result in the formation of water and further quantities of sodium sulphate so that the composition of the bath is continuously changing. It is customary therefore to withdraw some of the bath, either continuously or intermittently, to evaporate water from the bath and to crystallise Glauber's salt ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) from the bath after the evaporation; the crystallised salt is then separated from the liquor which is usually returned, if necessary after adjustment of its composition, to the coagulating bath. The Glauber's salt obtained contains small amounts of sulphuric acid and zinc sulphate and for economic reasons it has been the practice hitherto in the viscose industry to discharge the crystallised salt, together with the small amount of sulphuric acid and zinc sulphate associated with it, to waste.

It is known that Glauber's salt can be converted into anhydrous sodium sulphate, which has a higher commercial value than Glauber's salt, by heating it above its transition temperature (about 32.5° centigrade), either alone or in a saturated solution of sodium sulphate, the mother liquor being run off and cooled to provide further quantities of Glauber's salt.

The application of these processes to the viscose industry has not hitherto proved to be an economical commercial proposition. Thus the known proposals effect the conversion of a relatively small proportion of the total sodium sulphate in the Glauber's salt to the anhydrous salt since only about 16 parts of the 44 parts of Na_2SO_4 contained in 100 parts of Glauber's salt (parts being by weight) are converted to anhydrous sodium sulphate on heating Glauber's salt to its transition point. It is therefore only by repeatedly carrying out the steps of heating the Glauber's salt, separating the anhydrous sodium sulphate and cooling the saturated sodium sulphate solution simultaneously formed to crystallise out pure Glauber's salt, to be heated in turn, that the proportion of the total sodium sulphate in the crude Glauber's salt eventually converted to anhydrous sodium sulphate can be raised to a reasonable figure, for example, 60 per cent. Such repeated steps have been proved to be uneconomical.

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Again in the prior proposals the anhydrous sodium sulphate tends to deposit in hard, cake-like form on hot surfaces in the evaporators or heaters used, and also in certain circumstances in the pipes connecting the various pieces of apparatus used in the recovery process. This caking is believed to be mainly due to the fact that sodium sulphate has a negative solubility coefficient and consequently, heating sodium sulphate solutions from a low temperature, for example 32° centigrade to a high temperature, for example 80°–90° centigrade, causes deposition of sodium sulphate in the heaters.

In United States patent of Ebert No. 2,374,004 it is proposed to avoid entirely or to minimize and localize the effects of caking by using a concentrated solution of a sodium salt other than sodium sulphate, for example the chloride, bromide or nitrate, as the hot solution used to convert the crude Glauber's salt into anhydrous sodium sulphate.

The object of the present invention is to provide an improved, economic process for converting crude Glauber's salt into anhydrous sodium sulphate in the viscose industry.

In accordance with the present invention, a continuous process for the production of anhydrous sodium sulphate from Glauber's salt comprises continuously feeding the Glauber's salt into a stream of a saturated aqueous solution of sodium sulphate of gradually increasing temperature, the salt being introduced at a point where the temperature of the solution is below 32.5° centigrade, converting the Glauber's salt into anhydrous sodium sulphate by drawing it through the stream of solution into a heated zone of solution, that is a zone of solution maintained at a temperature above 32.5° centigrade, in a direction opposite to the direction of flow of the solution, which solution is continuously formed solely as a result of the heating of the Glauber's salt in the heated zone so that it thereafter flows continuously from the heated zone through the advancing Glauber's salt and so forms the stream of solution of gradually increasing temperature and, while cooling and flowing, it washes the Glauber's salt and also deposits pure Glauber's salt crystals, and continuously recovering the anhydrous sodium sulphate formed. The temperature of the solution at the point where the Glauber's salt is fed in is preferably about 12°–14° centigrade.

The present invention also includes apparatus for the production of anhydrous sodium sulphate from Glauber's salt comprising in combination,

an elongated vessel, mechanism for continuously feeding the Glauber's salt into one end of the elongated vessel, at least one mechanical device for drawing the Glauber's salt through the vessel from one end to the other, and heating means at the other end of the vessel to where the Glauber's salt is fed in, which heating means forms a heating zone to convert the Glauber's salt into anhydrous sodium sulphate and saturated sodium sulphate solution, the vessel being so arranged and constructed that the saturated sodium sulphate solution so formed flows towards the Glauber's salt feeding end of the vessel and forms a stream of solution the temperature of which gradually decreases as it flows through the vessel. In a preferred form of apparatus, cooling means is also provided from the beginning of the heated zone to the Glauber's salt feeding point so that the temperature of the flowing solution may be readily adjusted to give the desired temperature gradient. Both the heating and cooling of the vessel are preferably effected by jacketing the vessel and circulating either a heating fluid such as hot water or steam or a cooling medium such as water, as the case may be, through the jackets.

In one form of apparatus according to the invention, the vessel consists of a long tank or trough. The part of the tank or trough which forms the heating zone may be inclined if desired so that the washed crystals are allowed to drain before being converted into the anhydrous salt. In a further form of the invention, the conversion of the washed Glauber's salt to the anhydrous salt is effected in a heated container provided at the end of the tank or trough, and into which the salt is drawn, preferably after draining, by the mechanical device, the sodium sulphate solution which is simultaneously liberated being continuously fed back to the vessel at the beginning of the heated zone for example by an over-flow pipe. The pipe may be jacketed so that the temperature of the liquid can be adjusted to as near 32.5° centigrade as possible.

In a further form of apparatus according to the invention, the vessel is formed by a series of inter communicating circular tanks connected so that there is a step of the foot of each tank communicating with the succeeding tank. Each tank is provided with a rotating paddle which turns the Glauber's salt crystals round till they fall over the step into the next tank.

In carrying out the process according to the invention, the Glauber's salt may be drawn through the solution by an Archimedean screw or a system of rakes, paddles, scrapers or other mechanical devices. The mechanical devices used to draw the Glauber's salt through the solution may also be used to draw the anhydrous sodium sulphate into a suitable collecting device, for example a separating tank or a centrifuge, or a separate mechanical device may be used for this purpose. The liquor adhering to the crystals is separated by the centrifuge and if necessary is reheated and fed back into the stream of sodium sulphate solution at a suitable point.

The process according to the invention is preferably effected in a long tank or trough which may be of uniform cross-section throughout its length or may be formed by a series of intercommunicating circular compartments. The tank or trough may be straight or shaped as a circle for moving the rakes conveniently, with a short break between the point of entry of the crude Glauber's salt and the point of withdrawal

of the anhydrous sodium sulphate. The tank or trough in front of the heated zone is preferably immersed in a stream of cooling water moving in the same direction as the direction in which the Glauber's salt is raked. By a suitable adjustment of the dimensions of the tank, the rate of feeding and drawing of the Glauber's salt and the temperature and rate of flow of the cooling water, the process may be carried out so that anhydrous sodium sulphate is collected at one end of the tank while a cooled saturated solution of sodium sulphate of such low concentration runs away at the other end that it is possible continuously to recover about 85 per cent or more of the sodium sulphate in the crude Glauber's salt in the form of the anhydrous salt, depending on the temperature to which the solution is ultimately cooled.

In an alternative form of the invention, the tank extends so that the solution of sodium sulphate flows beyond the feeding point of the crude Glauber's salt so that the further cooling beyond this point for example to 12°-14° centigrade deposits substantially pure crystals of Glauber's salt and the crystals so deposited are recovered by drawing them by a second mechanical device in the same direction as the direction of the flow of the solution to a suitable collecting device such as a centrifuge. In this case, the point of addition of the crude Glauber's salt to the tank will be nearer to the heated zone of sodium sulphate solution than in the case in which only sodium sulphate solution is collected beyond the feeding point of the Glauber's salt and the temperature of the solution at the point of entry of the Glauber's salt will be correspondingly higher. This embodiment of the invention permits the simultaneous and continuous production of commercially pure anhydrous sodium sulphate and commercially pure Glauber's salt from crude Glauber's salt such as is obtained by evaporation and crystallisation of viscose spinning baths.

As a modification, sodium sulphate solution may be maintained by withdrawing part of the stream of solution at a point where its temperature is below 32.5° centigrade, heating the withdrawn solution and feeding it back into the stream at the point where the anhydrous sodium sulphate is recovered. The solution withdrawn in this manner is preferably reheated to a temperature at which the solubility of sodium sulphate in water is not below its solubility at the temperature at the withdrawal point. In this manner deposition of anhydrous sodium sulphate in the heater and in the pipes connecting the heater to the tank may be prevented. For example, if the solution is withdrawn at a temperature of 31.5° centigrade it may be heated to a temperature of 60° centigrade since the solubility of sodium sulphate in water is 31.2 per cent at both 31.5° centigrade and 60° centigrade. Information on the solubility of sodium sulphate is given by A. Seidell on page 1301 of his book entitled "Solubilities of Inorganic and Metal Organic Compounds," 3rd edition, volume 1, and published in 1940 by D. Van Nostrand Co. Inc. The withdrawn solution may also be treated to remove impurities such as sulphuric acid or zinc sulphate before it is returned to the tank.

When starting the process according to the invention it is not essential that there should be any saturated sodium sulphate solution in the tank since such solution will eventually be formed when the Glauber's salt reaches the heated zone but it is advantageous to have the

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tank or trough filled with a saturated solution as this solution will serve to wash some of the impurities from the crude Glauber's salt and so help to improve the purity of the anhydrous sodium sulphate initially precipitated in the heated zone. As soon as the tank has become filled with saturated sodium sulphate solution no further sodium sulphate solution is added to the tank as the solution will be subsequently provided continuously as the Glauber's salt is simultaneously transformed in the heated zone into anhydrous sodium sulphate and saturated sodium sulphate solution.

In the case of acid-contaminated Glauber's salt it may be desirable to neutralise the solution with alkali. Such neutralisation is preferably carried out in the neighbourhood of the heated zone.

In carrying out the process according to the invention it is important to establish a sodium sulphate solution of gradually increasing temperature and concentration in the tank or trough in front of the heated zone so that the highest temperature, which should be about 32.5° centigrade but not higher, will be immediately before the heated zone and the lowest temperature will be at the exit end of the tank where the cooled solution flows to waste.

It is also important that the action of the paddles or rakes should be so adjusted as to bring the crystals of Glauber's salt into substantial equilibrium with the solution at every stage of increasing temperature and concentration; this is particularly important at the last stage where the temperature is at or near to 32.5° centigrade for at this temperature the solubility of sodium sulphate is at its maximum. If therefore any of the solution is carried over with the Glauber's salt into the heating or decomposition zone, none or only a small proportion of the Glauber's salt is dissolved and a maximum yield of anhydrous sodium sulphate is attained. It is particularly in this respect that the process according to the invention differs from any of the known processes of converting Glauber's salt to anhydrous sodium sulphate for in the known processes it is usual to crystallise out the Glauber's salt at relatively low temperatures and therefore any adhering mother liquor when heated above the transition temperature of Glauber's salt is capable of dissolving some of the anhydrous sodium sulphate formed by the decomposition process thus decreasing the yield for a given consumption of heat. This effect may be shown by an actual example; if Glauber's salt is crystallised at, say, 12°-13° centigrade in accordance with standard practice and after separating the crystals these latter contain say 10 per cent of adhering mother liquor, then 111 grams of damp crystals would have contained 44 grams of Na_2SO_4 and 56 grams of water in the crystalline decahydrate and approximately 1.1 grams of Na_2SO_4 and 9.9 grams of water in the adhering solution. Therefore on heating these damp crystals to 32.5° centigrade at which temperature 100 grams of solution contain 33 grams of Na_2SO_4 and 67 grams of water, the 9.9 grams of adhering water would be capable of dissolving:

$$\frac{33 \times 9.9}{67} = 4.9 \text{ grams of } \text{Na}_2\text{SO}_4$$

of which only 1.1 grams are already dissolved in the solution.

Therefore instead of 100 grams of the decahy-

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drate yielding 16 grams of anhydrous sodium sulphate the yield would be reduced to:

$$16 - 4.9 + 1.1 = 12.2 \text{ grams}$$

That is the yield for a given consumption of heat used in the decomposition zone would be nearly 75 per cent of theory and nearly 25 per cent of the heat would be lost with an increasing load of like amount in the recrystallising zones.

In the process according to the invention however since the crystals are in equilibrium with saturated sodium sulphate solution at or near to the point of maximum solubility (32½° centigrade) just prior to entering the decomposition zone no or very very little sodium sulphate is dissolved in the adhering solution and therefore no especial care is necessary to dry the crystals between the crystallizing and the decomposition zones.

In accordance with a further feature of the present invention, the continuous production of anhydrous sodium sulphate, with or without the simultaneous production of pure Glauber's salt, is employed as part of a continuous process for the conservation and recovery of chemicals from viscose rayon spinning baths containing sulphuric acid, sodium sulphate and zinc sulphate. Thus spinning bath liquor may be withdrawn either continuously or intermittently and water evaporated from the liquor so that on cooling, Glauber's salt will crystallise out. In accordance with the invention, the cooling of the liquor, after the evaporation, is effected in a tank while the liquor is moving in a stream in the same direction as the rakes, scrapers or other device employed to move the Glauber's salt, and when the desired degree of cooling has been obtained, the mother liquor is allowed to drain away from the tank over a suitably placed weir and is returned to the spinning bath, if necessary after adjustment of its composition, while the crude crystals of Glauber's salt are continuously raked forward past the exit weir of the mother liquor into the moving stream of sodium sulphate solution. Thus, the crystallisation of the crude Glauber's salt from the evaporated liquor and also its conversion to anhydrous sodium sulphate may be effected in one long tank.

In order to prevent excessive contamination with acid of the saturated sodium sulphate solution flowing away from the heated zone it is desirable to slope the bottom of the tank upwards beyond the exit weir of the cooled evaporated acid solution in order to allow the Glauber's salt to drain a little before it is raked into the stream of saturated sodium sulphate solution moving in the opposite direction. The top of the slope should be above the level of the two solutions so that there is no tendency for the acid solution to mix with the other solution. After passing over the top of the slope the Glauber's salt is raked in the same direction along the tank into the heated zone where the anhydrous sodium sulphate is produced in accordance with the invention.

If desired the weak sodium sulphate solution containing any small quantities of acid and zinc sulphate which had adhered to the crude Glauber's salt may be passed over a weir of the tank and may be added to the mother liquor as it returns to the spinning bath.

The evaporated spinning bath liquor is preferably introduced into the tank at a temperature of the order of 40°-45° centigrade and cooled in

the tank to a temperature of about 12°–14° centigrade immediately before the weir.

The invention is illustrated by way of examples in the accompanying diagrammatic drawings in which

Figure 1 is a vertical section of an apparatus for converting Glauber's salt to anhydrous sodium sulphate in a long straight tank,

Figure 2 is an enlarged section on the line II—II of Figure 1,

Figure 3 is a vertical section of a modification of the apparatus of Figure 1 in which the washed Glauber's salt crystals are allowed to drain before being converted into the anhydrous salt,

Figures 4 and 5 illustrate a further modification in which a series of intercommunicating circular tanks is used, Figure 4 being a vertical section of the apparatus and Figure 5 being a plan view of part only of the tanks,

Figure 6 is a vertical section of an apparatus for producing both pure Glauber's salt and anhydrous sodium sulphate, and

Figure 7 is a vertical section of an apparatus for the continuous recovery of anhydrous sodium sulphate and other chemicals from viscose spinning baths.

Throughout the drawings, like reference numerals are included to indicate like parts.

Referring to Figures 1 and 2, crude Glauber's salt 1 is fed continuously from a hopper 2 into a long jacketed tank 3. A series of scrapers 4, carried by a bar 5, is continuously oscillated as shown so that the crystals within the tank 3 are slowly drawn from left to right through a stream of saturated sodium sulphate solution 6 flowing through the tank 3 from right to left and finally are drawn into the heated container 7 in which the washed Glauber's salt is converted into anhydrous sodium sulphate 8.

The tank 3 consists of two zones AB and BC, the zone AB being the cooled or crystallizing zone of gradually increasing temperature up to 32.5° centigrade and the zone BC being the heated or decomposition zone at a temperature above 32.5° centigrade, preferably about 40° centigrade. Higher temperatures may be used but are undesirable as they cause scaling on the container walls as a result of the negative solubility coefficient of sodium sulphate above 32.5° centigrade. Zone AB is provided with a baffled jacket 9 through which cooling water is continuously circulated, the water being fed in through an inlet pipe 10 and flowing out through an exit pipe 11. The heated zone BC has a surrounding jacket 12 which also surrounds the lower part of the container 7. Jacket 12 is heated by steam or hot water pipes 13, the heating medium flowing from pipe 14 to pipe 15 at such an initial temperature and at such a rate that at point B the temperature is about 32.5° centigrade.

The crude crystals 1 fed in at the end A are slowly drawn by the scrapers 4 through the stream of solution 6 and as they are advancing they are washed by the solution. At the point B the washed crystals enter the decomposition zone BC where their conversion into anhydrous sodium sulphate 8 and saturated sodium sulphate solution takes place. The crystals, as they fall to the bottom of the container 7, are stirred by the stirrer 16 so that a washing is effected; the stirrer 16 preferably has a very small clearance from the side of the container so that the crystals forming on the side are scraped off as they are formed. The saturated sodium sulphate solution formed then flows from the end C to-

wards the end A thus automatically and continuously forming the stream of solution 6. As the stream flows down the tank it is cooled after passing point B, where the temperature is regulated at or about 32.5° centigrade, and washes the advancing crystals and also deposits pure Glauber's salt to augment the crystals 1. At the same time its temperature gradually decreases as a result of cooling by the advancing crystals and the cooling water in jacket 9, the temperature and rate of flow of which is regulated to give the necessary temperature gradient having regard to the length of the tank. By the time the solution reaches point A its temperature will preferably be about 12°–14° centigrade and it then flows away through pipe 17 to waste; if desired the pipe 17 may be replaced by an open channel so that any crystals which become deposited are accessible for removal. When using acid-contaminated Glauber's salt it is desirable to neutralise the solution and this is preferably effected at point B just before the washed crystals enter the decomposition zone.

In the embodiment shown in Figure 3, the procedure is the same as that described with reference to Figures 1 and 2 with the following exceptions:

(a) In the decomposition zone BC the tank is inclined so that the washed crystals passing point B at or near 32.5° centigrade are allowed to drain before being decomposed. A separate set of oscillating scrapers 18 carried by a bar 19 is provided and is preferably oscillated in the same way and at the same speed as the scrapers 4.

(b) The drained crystals are fed into a separate container 20 to effect the decomposition. Container 20 is provided with a jacket 21, heated, for example to about 40° centigrade, by hot water or steam pipes 22.

(c) The saturated sodium sulphate solution formed as a result of the decomposition continuously overflows from container 20 and is fed by an open inclined channel 23 back into the tank 3 near to point B, the temperature of the solution at point B being approximately 32.5° centigrade when it enters the tank 3.

Instead of using scrapers 4 for drawing the Glauber's salt through the tank 3, a system of slowly moving rakes or an Archimedean screw system may be used.

Referring to Figures 4 and 5, the tank is formed by a series of intercommunicating tanks 24 having the desired temperature gradient from about 12°–14° centigrade at point A up to 32.5° centigrade at point B. The propulsion of the Glauber's salt crystals is carried out by a series of rotating paddles 25, one for each tank, which turn the crystals round until they fall over a step 26 into the succeeding tank. The paddles as shown are arranged to scrape the sides of the tanks to prevent undue scaling of crystals on the tank walls. The procedure is otherwise the same as that described with reference to Figures 1 and 2.

Referring to Figure 6, crude Glauber's salt 1 is fed from a hopper 2 into a jacketed tank 3 as described with reference to Figures 1 and 2 but in this case the hopper is arranged nearer to the heated zone of the tank so that the crude salt 1 is fed into the stream of solution at a higher temperature, for example about 20° centigrade and the tank 3 and the cooling jacket 9 are extended beyond the Glauber's salt feeding point, that is to the left of the hopper 2. The appara-

tus to the right of the hopper 2 for producing the anhydrous salt is not shown in full since it corresponds with that shown in either Figure 1 or Figure 3. The saturated sodium sulphate solution 6 after flowing past the hopper 2 continues to cool and in cooling deposits pure Glauber's salt crystals 27. These crystals are drawn by further systems of scrapers 28 and 29, in the same direction as the direction of flow of solution 6 into a container 30. The tank is sloped slightly at the end as shown so that excess liquid is removed from the pure Glauber's salt crystals and run to waste through a pipe 31. The cooling of the solution 6 to the left of the hopper 2 is preferably carried as low as possible for example down to 4° centigrade.

Referring to Figure 7, evaporated spinning bath liquor while still hot, for example at a temperature of 40° to 45° centigrade is fed from a pipe 33 into one end of a jacketed tank 34. The solution in the tank 34 begins to deposit crude Glauber's salt 35 which is drawn, together with its associated mother liquor, by a system of scrapers 36, carried by a bar 37, towards a weir 38. The crude Glauber's salt and mother liquor while being drawn along by the scrapers 36 are cooled by means of cooling water flowing from an inlet pipe 39 into a jacket 40, out through an exit pipe 41, and while so cooling further crystals of Glauber's salt are deposited. The temperature of the weir 38 is preferably about 12° to 14° centigrade. When the crude salt and the mother liquor reach the weir 38, they are drawn by an Archimedean screw 42 up and over the weir which is provided with holes or perforations 43 through which the mother liquor drains away down pipe 44. At the top of the weir the crude crystals are drawn by a second Archimedean screw 45 into a counter-current flow of sodium sulphate solution 6 where the crude salt is washed and converted into anhydrous sodium sulphate as described with reference to Figure 1 or Figure 3. The excess cold, saturated sodium sulphate solution at the weir 38 drains away through holes 46 into a pipe 47 and, as shown, is mixed with the mother liquor for return, if necessary after adjustment of the composition of the mixture, to the spinning bath.

What I claim is:

1. A continuous process for the production of anhydrous sodium sulphate from Glauber's salt which comprises heating Glauber's salt to a temperature above 32.5° centigrade to form anhydrous sodium sulphate and a saturated solution of sodium sulphate by passing the Glauber's salt longitudinally through a substantially horizontal tank having at one end a cold zone at a temperature below 32.5° C. and at the other end a hot zone above 32.5° C., continuously separating the saturated solution from the anhydrous sodium sulphate and recovering the anhydrous sodium sulphate by withdrawing it from the tank at the hot zone, causing the saturated sodium sulphate solution so formed to flow continuously as a confined stream along a substantially horizontal path away from the hot zone to the cold zone,

gradually cooling said stream of saturated solution so that its temperature gradually and uniformly falls to a temperature below 32.5° C. as it flows from the hot zone to the cold zone whereby the stream as it cools deposits Glauber's salt, continuously feeding additional Glauber's salt into said stream in the cold zone, continuously moving both the additional and the deposited Glauber's salt counter-current through said stream into the hot zone where the Glauber's salt is continuously converted to anhydrous sulphate and saturated sodium sulphate solution.

2. A continuous process for the production of anhydrous sodium sulphate from Glauber's salt which comprises heating Glauber's salt to a temperature above 32.5° C. in a hot zone to form a saturated solution of sodium sulphate and anhydrous sodium sulphate, causing the saturated sodium sulphate solution so formed to flow as a confined stream along a substantially horizontal path from the hot zone having a temperature above 32.5° C. to a cold zone at a temperature below 32.5° C. gradually cooling said stream of solution so that its temperature gradually and uniformly falls as it flows from the hot zone whereby the stream as it cools deposits Glauber's salt, continuously feeding additional Glauber's salt into said stream in the cold zone, continuously moving both the additional and the deposited Glauber's salt counter-current through said stream into the hot zone wherein the Glauber's salt is continuously converted to anhydrous sodium sulphate and saturated sodium sulphate solution, causing continuous flowing off of the saturated Na_2SO_4 solution from the cold zone and removing anhydrous sodium sulphate from the system at the hot zone.

3. A process as claimed in claim 2 wherein the temperature of the stream of solution at the point where the additional Glauber's salt is fed in is about 12 to 14° centigrade.

4. A process as claimed in claim 2 wherein the temperature of the stream of solution at the point where the additional Glauber's salt is fed in is about 20° centigrade.

HORACE J. HEGAN.

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