

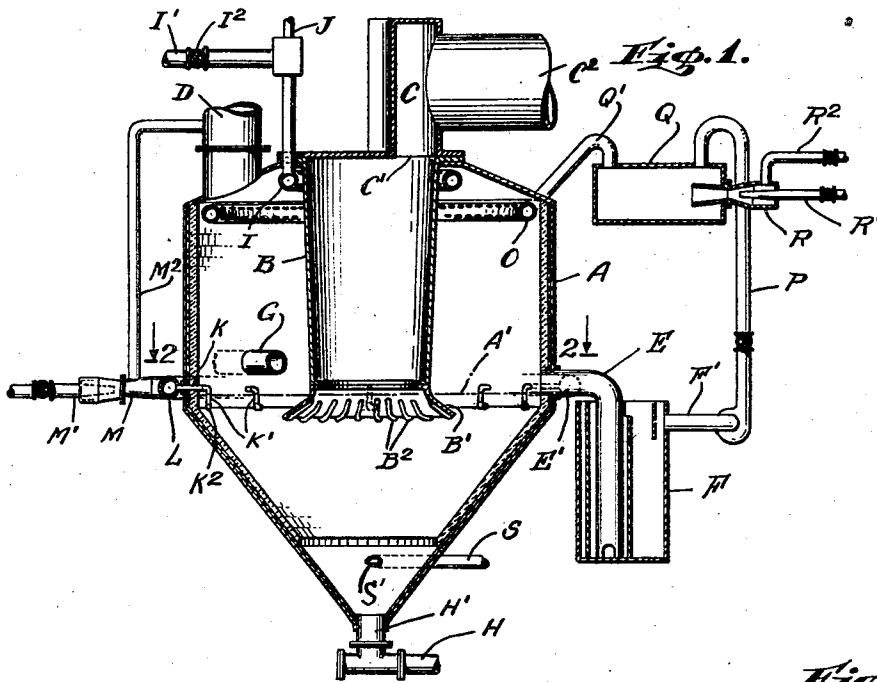
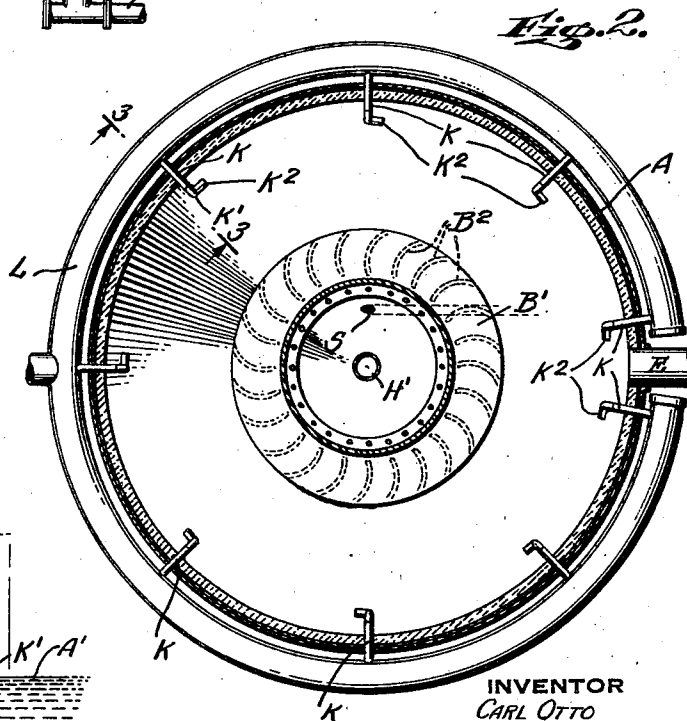
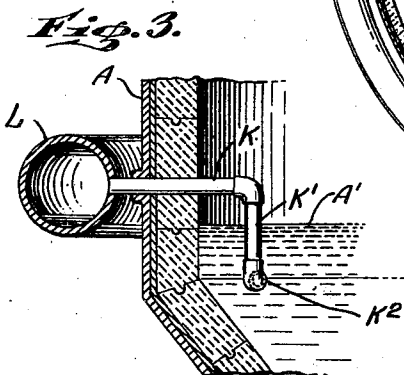
July 15, 1947.

C. OTTO

2,424,205

PRODUCTION OF AMMONIUM SULPHATE

Filed Feb. 10, 1943



INVENTOR
CARL OTTO

BY *John E. Hubbell*
ATTORNEY

UNITED STATES PATENT OFFICE

2,424,205

PRODUCTION OF AMMONIUM SULPHATE

Carl Otto, Manhasset, N. Y., assignor to Fuel Refining Corporation, New York, N. Y., a corporation of Delaware

Application February 10, 1943, Serial No. 475,371

5 Claims. (Cl. 23—119)

1

The present invention relates to apparatus for recovering ammonia from distillation gas by passing the gas through a so called saturator tank holding in its lower portion a weak sulphuric acid liquor bath with which the gas is brought into scrubbing contact with the resultant production of sulphate of ammonia which crystallizes out of the bath liquor and accumulates in the lower portion of the saturator, and from which the sulphate crystals are removed in a carrying stream of liquor. In practice, the scrubbing contact of the gas with the bath liquor is effected by passing the gas into the saturator through a so called cracker pipe which has a discharge mouth submerged in the bath liquor.

In the use of such a saturator, particularly when the bath liquor has the relatively low acid content required for the production of "large" crystals of sulphate of ammonia, troublesome sulphate of ammonia deposits, commonly called rock salt, form on the internal surface portions of the saturator. Such salt formations are wholly or mainly the result of the dehydrating action of the gas on drops and films of bath liquor splashed or otherwise deposited on portions of the saturator surface with which the gas comes into contact.

In my prior applications, Serial Nos. 473,949 and 473,951, filed January 29, 1943, I have disclosed arrangements for injecting water or other liquor into the saturator gas space which are effective to substantially minimize salt formations on the bulk of the internal saturator surface in contact with the gas passing through the saturator. I have discovered, however, that the arrangements described in my prior application are not always effective to prevent objectionable salt deposits on the portion of the inner wall of the saturator tank adjacent the bath level. Salt deposits on that portion of the tank wall are especially apt to occur in full load operation of a relatively large saturator with the relatively low bath acidity required for the production of large ammonium sulphate crystals, in consequence of the relatively violent splashing of the bath liquor against the portion of the saturator wall immediately above the bath level and the up and down movement of the top surface of the bath liquor in contact with the tank wall, resulting from the scrubbing contact of the gas and bath liquor.

A main object of the present invention is to provide novel and effective means for minimizing or preventing troublesome salt formation on the portion of the saturator tank wall adjacent the normal bath level. To attain this object, I lo-

2

cally heat the bath liquor immediately adjacent the portion of the tank wall intersected by the bath level. To thus heat the portion of the bath liquor adjacent said wall, I inject jets of steam into said portion of the bath liquor, by which the injected steam is condensed. By thus heating the bath liquor in contact with the tank wall adjacent the top of the bath, the capacity of that liquor to dissolve sulphate of ammonia and to hold the latter in solution is increased and that capacity is further increased by the local dilution of the bath liquor resulting from the addition thereto of the water of condensation formed.

To avoid water hammer effects, I advantageously mix compressed air or gas with the steam injected into the liquor. The addition of air or gas to the steam is beneficial, also, in that it results in an increase in the volume of the gaseous fluid injected into the bath liquor and thereby augments the scrubbing action of the bath liquor on the adjacent portion of the tank wall. While the addition of distillation gas is theoretically preferable to the addition of air to the steam injected into the bath liquor because the added gas does not change the composition of the gas passing away from the saturator, in practice the amount of air added to the steam to prevent water hammer is too small to have any significant effect upon the composition of the gas leaving the saturator.

The steam injected into the bath liquor to avoid objectionable salt deposits as above described, serves also to supply to the bath liquor in a relatively inexpensive and convenient manner heat which is needed to maintain a suitably high bath temperature notwithstanding the action of the gas treated in evaporating water out of the bath liquor and thereby cooling the latter.

Advantageously, the jets of steam and air or gas injected into the bath liquor are directed to create a continuous circular movement of the bath liquor around the axis of the saturator tank, or to augment the circular movement of the bath liquor about said axis which the gas passing out of the cracker pipe discharge mouth may produce when said discharge mouth is suitably shaped for the purpose.

A further object of the present invention is to provide improved means for preventing salt deposits on the saturator surfaces above and out of direct contact with the bath held in the lower position of the saturator.

The various features of novelty which characterize my invention are pointed out with par-

3

ticularity in the claims annexed to and forming a part of this specification. For a better understanding of the invention, however, its advantages, and specific objects attained with its use, reference should be had to the accompanying drawing and descriptive matter in which I have illustrated and described preferred embodiments of the invention.

Of the drawings:

Fig. 1 is an elevation in section of a saturator;

Fig. 2 is a plan in section on the line 2—2 of Fig. 1; and

Fig. 3 is an enlarged section on the line 3—3 of Fig. 2.

In the drawings I have illustrated an embodiment of the present invention in a saturator comprising a tank A of conventional form and construction, provided with a vertical centrally disposed cracker pipe B having a separately formed discharge mouth piece B' suitably attached to its lower end and submerged in the bath liquor filling the lower portion of the tank to its overflow level A'. The cracker pipe B receives gas at its upper end through the outlet C' from a gas inlet chamber C receiving gas from a supply main C². The gas flowing downward through the cracker pipe passes into scrubbing contact with the bath liquor and thence upward away from the bath into the annular gas space above the bath level A' and surrounding the cracker pipe B, and passes away from that gas space through the gas discharge pipe D. The bath liquor level A' is determined by the position of a weir E', advantageously adjustable, in a bath liquor overflow outlet E. As shown, the latter comprises a depending conduit portion extending down into a tar separator F of known type including a chamber from the top of which tar may be skimmed or otherwise removed. The separator F has an outlet F' for the tar free bath liquor. Bath liquor withdrawn from the saturator may be returned through a pipe G and also through other pipes Q' and S hereinafter referred to.

Ammonium sulphate crystals accumulating in the lower portion of the bath are withdrawn from the latter in a carrying stream of bath liquor by suitable means shown as comprising a pipe H, having an inlet branch H' communicating with the tank at the apex of its hopper bottom portion. Customarily, the pipe H leads to the inlet of a suitable pump (not shown), by which the bath crystals and the crystal enveloping bath liquor are delivered to suitable crystal drying apparatus. As shown, a spray pipe ring I surrounds the cracker pipe adjacent its upper end and is adapted to spray liquid against the inner tank wall and external cracker pipe surfaces, to thereby wash off sulphate of ammonia splashed against the said surfaces from the bath, and passing out of entrainment in the gas coming into contact with said surfaces as it passes upward from the bath to the gas outlet D. The wash liquid is supplied to the spray pipe I through a supply pipe I' at a rate which may be regulated by the adjustment of a valve I². The wash liquid supplied to the spray ring I may well be hot water though in some cases it may be bath liquor, and in either event may include make-up acid which must be supplied to the bath to make up for the acid converted into sulphate of ammonia. As shown the make-up acid is supplied through a pipe J discharging into the supply pipe I.

In accordance with the present invention bath heating fluid is injected into the bath adjacent

4

its periphery and a few inches below the bath level A', through a multiplicity of pipes K distributed about the tank axis. Each pipe K in the form shown in the drawings, comprises a horizontal portion extending through the saturator tank wall and connected at its outer end to a manifold L extending circularly about the saturator tank B. Each spray pipe K comprises a down-turned portion K' within the tank and a horizontal discharge end portion K² extending substantially at right angles to an adjacent plane including the common axis of the cracker pipe B and tank A.

The heating fluid injected may advantageously be supplied at a pressure of only a pound or so above atmospheric pressure. As shown in Fig. 1, the fluid supplied to the manifold L is steam mixed with distillation gas by means of a steam injector M to which steam is supplied by a pipe M' and having a mixing chamber receiving gas from the saturator gas outlet D through a pipe M².

As shown, the cracker pipe mouth piece B' is outwardly flared and is provided at its underside with ribs B² which extend inward from the rim of the mouthpiece and are curved, as shown in Fig. 2, two discharge gas streams each having a substantial flow component in a direction tangential to the rim of the mouthpiece. In consequence said gas streams create a circulatory movement of the bath liquor adjacent the top of the bath about the saturator axis which is in the clockwise direction when the ribs B² are curved as shown in Fig. 2. Advantageously, and as shown, the discharge ends K² of the pipes K are so directed relative to the direction in which the outer ends of the ribs B² diverge from planes radial to the cracker pipe axis that the bath liquor circulation induced by the pipe ends K² and that induced by the gas streams defined by the curved ribs B², are in the same direction, which as shown in Fig. 2, is clockwise.

In the contemplated mode of use of the present invention steam is supplied by the pipe M' at such temperature and in such volume relative to the saturator bath volume and temperature, that substantially all of the steam injected into the bath liquor is condensed therein and the portion of the bath liquor into which the steam is injected is thereby maintained at a temperature somewhat above the average temperature of the saturator bath.

By thus heating the bath liquor in contact with the tank wall adjacent the top of the bath, the capacity of that portion of the bath liquor to hold sulphate of ammonia in solution is appreciably increased, and the possibility that crystallized sulphate will deposit on the portion of the tank wall by such liquid is correspondingly reduced. The possible occurrence of such deposits is further reduced as a result of the dilution of the portion of the bath liquor into which the steam is injected by the water of condensation formed, and is further reduced by the scrubbing action on the wall of the bath liquor due to its agitation by the jets discharged through the pipes K. The gas mixed with the steam discharged through said pipes serves to increase the volume and scrubbing effect of the jets in addition to serving its primary purpose of preventing water hammer vibration noise.

As previously explained, the heat added to the bath by the steam injected into the latter raises the average bath temperature and thus reduces the quantity of heat which must be constantly added to the bath, in many cases, to avoid an

unduly low temperature of the gas liquor which is subjected to the cooling effect of the evaporation of the water out of the bath by the gas passing through the saturator.

While the injection of hot water into the saturator through the spray pipe ring I at a suitable rate will substantially retard the formation of sulphate deposits upon the upper portions of the inner surface of the saturator wall and on the outer surface of the cracker pipe, it is not practically feasible to supply enough wash water through the spray pipe I to prevent sulphate deposits on the exposed upper surface of the saturator, without risk of "killing" the bath, i. e., of reducing the acid content of the gas to an extent which prevents sulphate of ammonia from crystallizing out of the bath liquor.

In the construction shown in Fig. 1, the means for avoiding sulphate deposits on the upper saturator surfaces comprises a spray pipe O as well as the previously mentioned spray pipe I. The pipe O extends circularly about the axis of the saturator adjacent the upper end of the inner side of the cylindrical body portion of the tank wall. As shown, heated bath liquor is supplied to the spray pipe O by means of a pump P which draws tar-free liquor from the tar separator F through its outlet pipes F' and discharges the bath liquor through a pipe P' into a suitable heater Q from which the heated bath liquor passes through a pipe Q' to the spray pipe O. In the simple and relatively inexpensive form illustrated, the heater Q comprises an enclosed chamber through which the liquor received from the pipe P' passes to the discharge pipe Q', and in which the liquor is heated by direct contact with a mixture of steam and non-condensable gas passed into the chamber through a tubular element R. The latter, in the simple form shown somewhat diagrammatically in Fig. 1, comprises a mixing chamber open at one end to the heating chamber and at its other and closed end, receives steam through a supply pipe R' and a non-condensable gas through a pipe R².

The non-condensable gas supplied by the pipe R² may be compressed air or distillation gas. As will be apparent, the liquor heating operation effected by the steam and air mixture discharged into the heater Q through the element R is identical in principle with that effected by discharging a steam and gas mixture into the saturator bath through the pipes K. In each case the steam is condensed in, and the water of condensation formed is mixed with the liquor heated, and in each case non-condensable gas mixed with the steam substantially prevents the water hammer shock or vibration and noise commonly experienced when steam is discharged into the water at a lower temperature through a submerged nozzle.

The effect of heating the gas liquor passing through the spray pipe O is to increase the capacity of the liquor to hold sulphate of ammonia in solution. It is practically feasible to supply heated bath liquor through the pipe O in the manner described in such volume and at a temperature sufficiently above the saturator bath temperature to entirely prevent sulphate deposits on the saturator surfaces washed by that liquor while also supplying enough steam to the saturator through the pipes K for their intended operative effect without risk of an objectionable dilution of the bath or an objectionable increase in the bath temperature. As will be apparent, the amount of bath liquor sprayed into the spray pipe

O will ordinarily have no significant effect on the strength of the saturator bath.

As will be apparent, the supply pipe I may also be supplied with bath liquor through the heater Q. In many cases, however, it will be practically convenient and desirable to supply water, preferably hot, to the spray pipe I, in amounts sufficient to prevent sulphate deposits on the outer surface of the cracker pipe and on the portion of the saturator tank roof adjacent the spray pipe O, while supplying heated bath liquor to the spray pipe O in amounts sufficient to prevent rock salt deposits on the inner saturator wall surfaces washed by the spray from the pipe O. Advantageously, and as shown, the connection flange B³ on the adjacent ends of the cracker pipe body and mouth piece B' are at the inner side of the cracker pipe and mouth piece, so that the outer surfaces of the cracker pipe and its mouth piece are smooth and oppose no obstacles to the maintenance of a continuous downflowing film of the wash water discharged against the upper end of the cracker pipe by the spray pipe I.

As shown in Fig. 1, the pipe S returns bath liquor to the saturator through a tangential inlet S' formed in the hopper bottom portion of the saturator tank wall. The liquor returned to the saturator through the inlet S' may come from any one or more of the usual sources of liquor withdrawn from the saturator. Preferably, the rate at which liquor is returned through the inlet S' is sufficiently in excess of the rate at which liquor is withdrawn from the saturator through the pipe P, so that there is an appreciable flow of liquor upward from the level of the inlet S'. The circularly and upwardly directed flow of liquor in the lower portion of the saturator due to the liquor return through the inlet S', creates a barrier or baffle which materially minimizes the gravitational downflow to the pipe H of fine sulphate crystals, without offering similarly minimizing opposition to the downflow of the larger crystals. As shown in Fig. 1, the usual brick lining for the lower portion of the metallic saturator tank wall, is cut away to provide a smooth walled space of suitable size for the circulation created by the return of liquor through the inlet S'.

Novel features disclosed herein and operating to prevent fine crystals from moving freely with the larger crystals to the inlet of the crystal withdrawing conduit H, which are disclosed but not claimed herein, are claimed in my copending application Serial No. 492,265, filed June 25, 1943.

While in accordance with the provisions of the statutes, I have illustrated and described the best forms of embodiment of my invention now known to me, it will be apparent to those skilled in the art that changes may be made in the forms of the apparatus disclosed without departing from the spirit of my invention as set forth in the appended claims, and that in some cases certain features of my invention may be used to advantage without a corresponding use of other features.

Having now described my invention what I claim as new, and desire to secure by Letters Patent is:

1. In the production of sulphate of ammonia crystals in a saturator in which a bath of sulphuric acid is maintained at the bottom of the tank and a gas space above the bath, the method which comprises passing ammonia containing gas

through said saturator and into scrubbing contact with the bath liquor therein and thence through said gas space, and minimizing salt deposits on the portion of the tank wall adjacent the level of its intersection with the top surface of the bath by heating the portion of the bath immediately adjacent said wall to a temperature above the average temperature of the bath by injecting steam into said portion of the bath.

2. In the production of sulphate ammonia crystals in a saturator in which a bath of sulphuric acid is maintained at the bottom of the tank and a gas space above the bath, the method which comprises passing ammonia containing gas through said saturator and into scrubbing contact with the bath liquor therein and thence through said gas space, and minimizing salt deposits on the portion of the tank wall adjacent the level of its intersection with the top surface of the bath by heating the portion of the bath immediately adjacent said wall to a temperature above the average temperature of the bath by injecting into the portion of the bath thus heated steam admixed with a non-condensable gas which minimizes vibration and noise resulting from the condensation of the steam.

3. In the production of sulphate of ammonia crystals in a saturator in which a bath of sulphuric acid is maintained at the bottom of the tank and a gas space above the bath, the method which comprises passing ammonia containing gas into scrubbing contact with the bath liquor in the central upper portion of the bath and thence into and through said gas space in such manner as to create a circular movement of said bath liquor about a vertical axis extending centrally through the tank and injecting heating fluid jets into the upper portion of the bath liquor adjacent the tank wall to thereby heat the last mentioned bath liquor to a temperature above the average bath temperature and to augment the circulation of bath liquor about said axis.

4. Apparatus for producing sulphate of ammonia by scrubbing contact of distillation gas with sulphuric acid liquor comprising in combination a saturator shell adapted to contain a bath of sulphuric acid in the lower portion thereof and having a withdrawal pipe constructed and located so as to establish an intermediate level of liquid therein, a vertically disposed pipe extending downwardly into said shell and having a flared

mouth below said intermediate level, inlet means for gases into said pipe, and liquid heating means within and extending about the axis of the shell and disposed in an annular portion of the space therein adjacent the wall of the said shell and below but adjacent said intermediate level.

5. Apparatus for producing sulphate of ammonia by scrubbing contact of distillation gas with sulphuric acid liquor comprising in combination a saturator shell adapted to contain a bath of sulphuric acid in the lower portion thereof and having a withdrawal pipe constructed and located so as to establish an intermediate level of liquid therein, a vertically disposed pipe extending downwardly into said shell and having a flared mouth below said intermediate level, inlet means for gases into said pipe, a liquid heating means comprising conduit means within said shell and having outlet openings for the discharge of a heating fluid distributed about the axis of said shell and located in an annular portion of the space within the shell and adjacent the wall of said shell and inlet means for the passage of a heating fluid into said conduit means.

CARL OTTO.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,932,674	Pyzel	Oct. 31, 1933
2,249,192	Titlestad	July 15, 1941
1,936,308	Mueller	Nov. 21, 1933
1,972,730	Connell	Sept. 30, 1934
2,067,043	Caldwell	Jan. 5, 1937
2,055,211	Yoder	Sept. 22, 1936

FOREIGN PATENTS

Number	Country	Date
507,351	Great Britain	June 14, 1939
361,935	Great Britain	Nov. 23, 1931
16,193	Great Britain	Oct. 20, 1910
639,041	Germany	Nov. 27, 1936
648,540	Germany	Aug. 3, 1937

OTHER REFERENCES

Bell, "Methods for the Improvement of By-Product Sulphate of Ammonia," printed in the Gas World, December 3, 1938, page 18.