Oct. 30, 1945. F. WETHLY 2,387,818

APPARATUS FOR THE PRODUCTION OF SULPHATE OF AMMONIA

Filed June 28, 1941

Fig. 1.

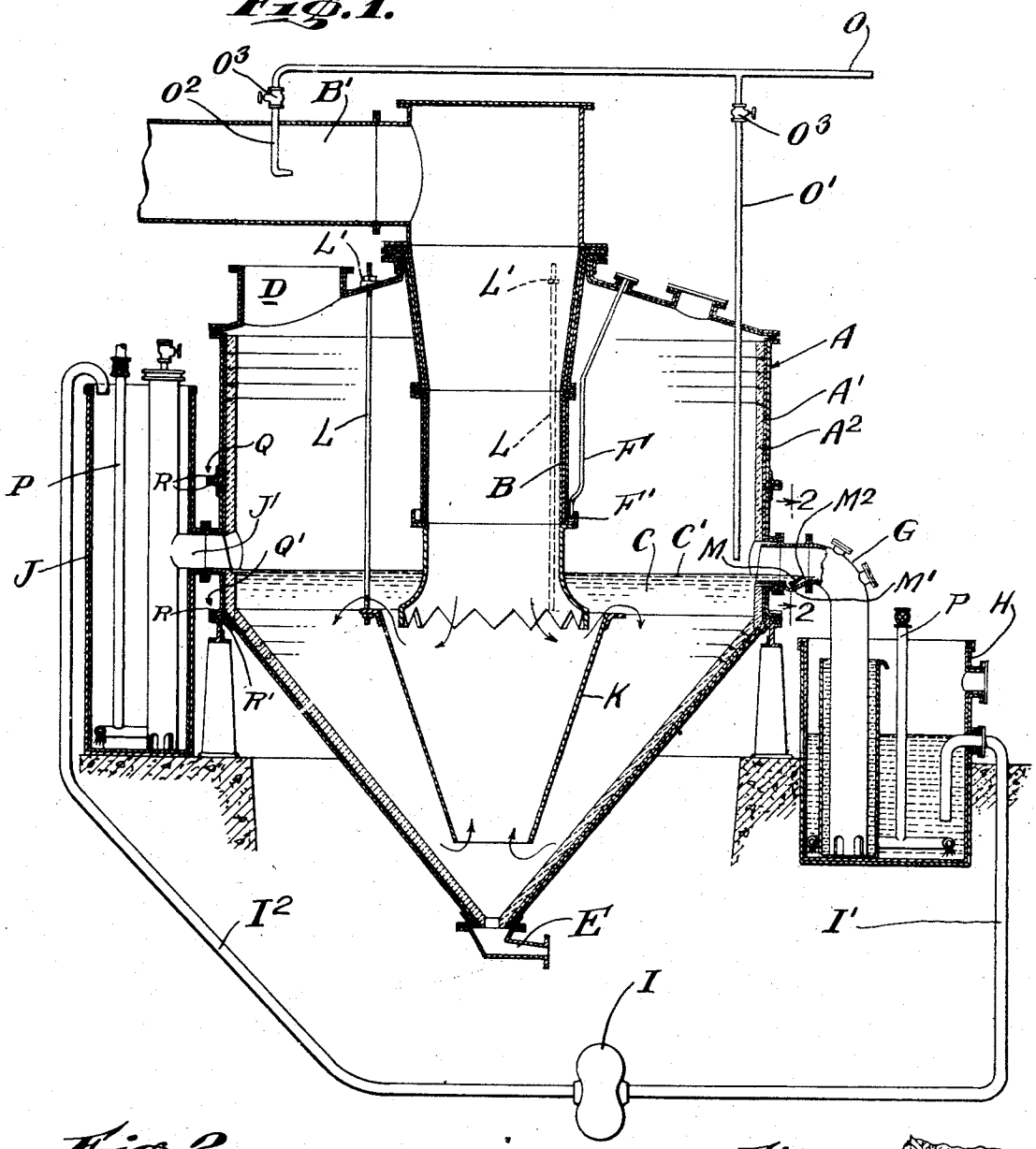


Fig. 2.

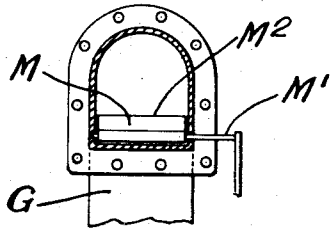


Fig. 3.

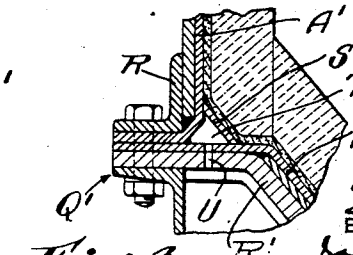
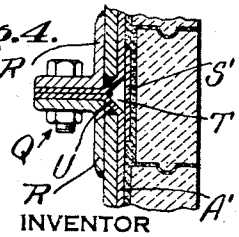


Fig. 4.



INVENTOR
Franz Wethly
BY
John E. Hubbell
ATTORNEY

Patented Oct. 30, 1945

2,387,818

UNITED STATES PATENT OFFICE

2,387,818

APPARATUS FOR THE PRODUCTION OF SULPHATE OF AMMONIA

Frans Wethly, Manhasset, N. Y., assignor to Fuel Refining Corporation, Dover, Del., a corporation of Delaware

Application June 28, 1941, Serial No. 400,329

1 Claim. (Cl. 23—273)

The general object of the present invention is to provide an improved method of and improved apparatus for recovering the ammonia content of distillation gases, and particularly of coke oven gas, in the form of sulphate of ammonia.

Sulphate of ammonia is customarily produced from coke oven gas by passing the gas into a so-called saturator containing a weak sulphuric acid bath through which the gas is bubbled, with the result that the ammonia in the gas and the sulphuric acid radical combine to form sulphate of ammonia, which crystallizes out of the bath solution.

A specific object of the present invention is to improve the above described process in such manner as to effect the production of sulphate crystals of relatively large size, while at the same time avoiding the rock salt formation difficulties heretofore experienced in producing sulphate crystals of relatively large size.

In accordance with the present invention, I form desirably large crystals in a manner heretofore known, by employing a bath of relatively low acidity, and by continuously maintaining a considerably greater amount of sulphate crystals in the bath than is customary in the production of crystals of the ordinary size, and by agitating the bath liquid, so that the small crystals initially formed are given time and opportunity to grow.

While it is theoretically possible to effect the agitation of the bath liquid for the above described purpose by injecting into the bath liquid, air or gas supplied at considerable pressure, or by the use of mechanical bath recirculating provisions, a specific object of the present invention is to provide a novel method of and means for utilizing the kinetic energy of the distillation gases bubbled through the bath to effect the desired bath agitation.

In its preferred form, the means devised by me for agitating the bath liquid, comprises a hollow open-ended recirculating element immersed in the bath liquid and open at top and bottom and in telescopic relation with the lower end of the usual dip or "cracker" pipe and having its lower end extending down into the bath liquid. Preferably, also, my improved apparatus includes means for varying the relative levels of the bath liquid, of the lower end of the dip pipe, and of the upper end of the recirculating or baffle element.

For the production of large crystals in the manner described, the saturator bath needs to have the relatively low acidity corresponding to a free acid content of from three to five per cent. With a higher acid content the sulphate dissolving capacity of the bath neutralizes the tendency to crystal growth produced by agitating the bath and prolonging the period of crystal retention in the bath.

The use of a bath having the relatively low acidity required for the production of large crystals has heretofore been attended by seriously objectionable deposits of crystallized sulphate on the internal walls of the saturator above the bath. Such deposits, commonly called rock salt, comprise stalactite-like masses which, in a comparatively short time become large enough to objectionably choke or block the gas flow through the saturator. Such rock salt formation results from the dehydration by the gas, of the drops and films of bath liquid adhering on and flowing along the saturator internal walls, onto which there is constantly being deposited liquid splashed, or carried out of the bath by entrainment in the gas bubbling through and rising above the bath. Such bath liquid deposits do not result in objectionable rock salt formations when the bath's acidity is higher, since, in such case, the dehydrated residue of previously deposited liquid is constantly being redissolved and washed off the saturator walls by fresh bath liquid deposits.

I have discovered that it is practically feasible to eliminate, or substantially minimize, objectionable rock salt deposits in a saturator operating with a bath acidity low enough for the production of large ammonium sulphate crystals, by adding steam to the gas as required to increase the moisture content of the gas in the saturator to the saturation point, and to maintain a saturator temperature of about 45° to 60° C. When the gas passing upward from the saturator bath is saturated with water vapor, it has no tendency to dehydrate the bath liquid drops or films on the saturator walls, and with a saturator temperature of 45° to 60°, the desired bath strength may be readily maintained.

Another specific object of the invention is to desirably minimize the amount of entrained bath liquid carried out of the saturator by the gas leaving the latter. Heretofore it has been attempted in some cases to provide baffles within the saturator gas space whereby gas bubbling out of the bath liquid passes through the saturator gas outlet along a path of flow more or less tortuous and at a velocity necessarily increased by the tortuousness of the flow path. I have discovered that better results are obtainable by omitting the baffles and by permitting the gas to flow directly from the bath liquid to the saturator

gas outlet with a suitably low velocity, and by making the flow path from the bath liquid to the gas outlet long enough to provide sufficient time for the entrained liquid to fall out of entrainment and back into the bath.

In the practical attainment of the above mentioned objects of my invention it is practically feasible and desirable in some cases, at least, to make use of a saturator of greater volumetric capacity than has heretofore been customary. While the construction of such a saturator may follow the general practices of the prior art, a specific object of the present invention is to provide an improvement in the lead lining of the saturator which is of especial importance in the production of saturators of relatively large size.

The various features of novelty which characterize the present invention are pointed out with particularity in the claim 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 in which I have illustrated and described a preferred embodiment of the invention.

Of the drawing:

Fig. 1 is a sectional elevation of a saturator embodying the novel apparatus features of the present invention and adapted for use in the practice of my improved method;

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

Figs. 3 and 4 are reproductions on a larger section of different portions of the apparatus shown in Fig. 1.

The apparatus shown in the drawing comprises a saturator tank A having a cylindrical body portion and a conical or hopper bottom portion. The saturator is formed in a known manner with an outer steel plate wall, an intermediate lead lining A' and an inner lining for its body and bottom portions formed by acidproof bricks A^2 having interlocking tongues and grooves. The distillation gas from which ammonia is to be recovered passes into the saturator through an axially disposed dip pipe B depending from the top wall of the saturator and having an open, outwardly flared, lower end located a foot or so below the normal top level C' of the bath liquid C. The cracker pipe B receives gas at its upper end from a gas supply pipe B' and the gas passing down through the cracker pipe into the bath liquid bubbles up through the latter and passes out of the saturator tank through the gas outlet D formed in the saturator top wall. Acid may be added to the bath as required by an acid supply pipe F discharging into an annular overflow trough F' surrounding the cracker pipe B some distance above the bath liquid level C'.

The conical or hopper bottom of the tank A is provided at its lower end with an outlet E adapted to be connected to a pump or analogous device, not shown, for withdrawing sulphate of ammonia crystals from the tank.

To prevent objectionable tar accumulations in the bath liquid the latter is allowed to overflow more or less continuously through an overflow outlet pipe connection G, which extends downwardly into an external bath liquid purifying vessel H. The purified gas liquid is withdrawn from the purifier H by a circulating pump I having its inlet I' connected to the purifier, and having its outlet I^2 discharging into a purified bath liquid reservoir J. The latter has an overflow outlet J' through which bath liquid is returned more or less continuously to the saturator.

In accordance with the present invention a recirculation of the bath liquid to increase the size of the sulphate crystals is effected by associating a recirculating baffle or element K with the cracker pipe in such manner that the kinetic energy of the gas discharged into the upper portion of the bath from the cracker pipe B will cause the bath liquid to flow up in a central portion of the tank directly beneath the discharge end of the cracker pipe B and downwardly through an outer portion of the tank surrounding said central portion. In the preferred form shown, in the drawing, the baffle or recirculating element K employed, is a hollow, open-ended body in the form of a conical frustrum having its large diameter end uppermost and in telescopic relation with the lower end of the cracker pipe and at a level intermediate the lower end of the latter and the bath liquid level C'. The lower end of the element K may well be at a level substantially below the liquid level C' but displaced upward from the lower end of the hopper bottom of the tank to provide a suitable space in which any crystables may collect preparatory to their withdrawal from the outlet E.

As will be apparent the gas passing down into the bath liquid from the cracker pipe B, reverses its direction of flow and passes upwardly through the annular telescopic joint space between the lower end of the cracker pipe B and the upper end of the element. As indicated by the arrows in Fig. 1, a portion of the kinetic energy of the gas entering the bath is imparted to the bath liquid which is thereby moved upward through said telescopic joint space and then outwardly over the upper edge of the member K. The kinetic energy of the gas thus creates a bath liquid upflow through the member K and a corresponding downflow through the annular portion of the bath surrounding the member K.

While the present improvements are adapted for use in saturators varying widely in form, dimensions and capacity, I note by way of illustration and example, that the particular saturator design reproduced in the accompanying drawing, has a tank diameter of twenty feet, and that the bottom of the element K is a couple of feet or so above the bottom oule of the tank. The element K may be rigidly mounted in the tank structure in any suitable manner. Preferably, however, the element K is vertically adjustable, and as illustrated, said element is supported by suspension bolts L having their threaded ends extending upward through the tank roof and engaged by nuts L' which may be rotated to adjust the elevation of the element K. The weight of the latter may be relatively small. It may be formed, for example, of steel plate one-eighth of an inch thick and lead coated, or it may be formed by a somewhat thicker but uncoated Monel metal plate or acid-proof bronze plate.

Even though the element K is made vertically adjustable as described, I advantageously provide means for adjusting the level C' of the bath liquid relative to the lower end of the dip pipe. This adjustment is provided for in the construction shown, by mounting a weir or dam element M in the horizontal portion of the bath liquid outlet pipe G, to turn about a horizontal axis M' and thereby raise or lower the free edge M^2 of the member M. The said edge M^2 serves as a weir edge over which the escaping bath liquid flows.

The angular adjustment of the dam or weir element thus determines the height of the bath liquid level C'.

For the purposes of the present invention, the gas in the saturator gas space between the bath level C' and the outlet D, may be saturated with water vapor either by injecting all or a portion of the steam required for the purpose directly into said space, or into the gas supply piping B' through which the distillation gases pass to the cracker pipe. As shown in Fig. 1, steam may be passed directly into the saturator gas space through a branch O' from the steam supply pipe O, and steam may be passed into the gas piping B' through a branch O^2 from the pipe O. The amount of steam discharged from each branch pipe may be regulated by a corresponding regulating valve O^3. As shown, pipes P are provided to supply hot wash water when needed to the vessels H and J.

In normal practice, the coke oven gas approaching the inlet B' prior to its exit of steam from the pipe Q^2, will be saturated and at a temperature of 35° to 40° C., as a result of its previous washing and cooling treatment. In the preferred mode of practice of the present invention, the steam supplied by the pipe O and added to the gas is adequate in amount and temperature to increase the temperature of the gas to 45° to 60° C., and to maintain the saturation of the gas as its temperature is so increased. As will be apparent, the rate at which steam is thus added to the gas may be regulated automatically or manually in response to gas condition measurements or other indications of saturator performance.

In the particular saturator design shown in the drawing, the vertical length of the cylindrical body portion of the saturator shell is ten feet and, as previously stated, the diameter of the shell is twenty feet. Under normal load conditions and with those saturator dimensions, the velocity of gas flow downward into the bath through the cracker pipe B will be of the order of 30 to 40 feet per second, and the velocity of the vertical component of the gas flow upward through the substantially unobstructed saturator gas space, from the bath liquid to the saturator gas outlet D, will be of the order of from two to three feet per second. With that relatively low velocity and with the outlet D eight feet or so above the bath liquid, a period of three or four seconds is required for the movement of the gas from the surface of the bath to the outlet D. With a period for the gas flow from the saturator outlet of the order of three or four seconds, most of the bath liquid entrained in the gas bubbling up out of the bath drops out of the gas before the latter reaches the outlet and hence falls back into the bath. In consequence, the entrained bath liquid carried out of the saturator is relatively small and can be recovered in a relatively small acid catcher or trap which may be of well known form and hence need not be illustrated or further described herein.

With a saturator of the unusually large dimensions mentioned above, it is ordinarily advantageous to divide the vertical body portion of the saturator shell into upper and lower sections united by a horizontal joint connection Q. The joint connection Q shown comprises connecting or clamping rings R, each of angle bar cross section with one flange horizontal, transverse to, and extending outwardly away from the axis of the saturator, and with its other flange alongside and welded to the corresponding section of the metallic body of the saturator shell. The side by side transverse lead sheets forming the sections of the lead lining A' respectively above and below the joint have outturned flange portions at their adjacent ends which are clamped between the transverse flanges of the rings R by means of the corresponding clamping bolts. The joint Q' between the lower end of the cylindrical body portion of the saturator shell and the conical bottom portion of the shell is generally similar to the above described joint Q. In the joint Q, however, the lower clamping ring member R' comprises a conical flange fitting against the outer surface of the conical metallic body portion of the shell and welded thereto. As shown, the upper end of the bottom portion of the shell is of smaller internal diameter than the superposed cylindrical portion of the shell, and the horizontal flange of the ring R' extends inwardly accordingly.

With the relatively large saturator dimensions mentioned above, and the considerable differences between the working temperatures and the cold temperature of the apparatus, the lead lining is subjected to substantial thermal expansion and contraction stresses which may have injurious effects on the lining at its joints. To avoid difficulty from those causes I connect spaced apart portions of the lead lining sections meeting at the joint Q by a continuous strip or rim of sheet lead S in the form of a frustum of a hollow cone which is welded, or "burned," at its upper and lower edges to the main lead lining sections extending in opposite directions away from the joint Q. With the main lining parts adjacent the joint Q connected by the ring S as described, an annular space T is formed in the lining at the outer side of the ring S. To avoid difficulties which otherwise might result from the thermal expansion and contraction of air in the space T, I connect that space to the atmosphere by one or more vent passages or "breather" openings U. The opening U shown in Fig. 3 extends vertically through the horizontal flange of the clamping ring R' and through the horizontal portion of the lead lining A' directly above that flange.

As shown in Fig. 4, a lead ring S' of cylindrical form is welded or "burned" at its upper and lower edges to the corresponding sections of the main lead lining A' extending upwardly and downwardly, respectively, from the joint Q. The air space T at the outer side of the sealing ring S' is vented to the atmosphere, as shown in Fig. 4, by an upwardly inclined passage U extending through the corner portion of the lower clamping ring R and through the corresponding portion of the lead lining A'.

No lead sealing strip analogous to the strips S and S' shown in Figs. 3 and 4 is provided at the joint between the upper end of the cylindrical and roof sections of the saturator shell, since it is practically essential that the roof section of the shell, should be readily separable from the subjacent portion of the shell to facilitate inspection and repairs. With the roof section readily separable, the joint between it and the subjacent portion of the shell may readily be sealed by a gasket such as is customarily employed at that joint.

While in accordance with the provisions of the statutes I have illustrated and described the best form 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 form of

the apparatus disclosed without departing from the spirit of my invention as set forth in the appended claim 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:

A saturator for the production of ammonia sulphate from coal distillation gas comprising a tank including an outer shell formed in sections separated by a horizontal joint and including an acid-proof lining comprising upper and lower lead lining parts respectively extending upward and downward from said joint and including side by side outwardly extending flanges clamped together at said joint, and a horizontally extending lead strip at the inner side of said joint and having its upper and lower edges integrally connected to said upper and lower lining parts respectively, said tank, strip and lining parts being shaped to provide an air space at the outer side of said strip and between it and said lining parts and a vent opening connecting said air space to the external atmosphere.

FRANS WETHLY.