E. E. ARNOLD.

PROCESS FOR RECOVERING CYANIDS AND THE LIKE FROM CYANID BEARING MATERIAL.

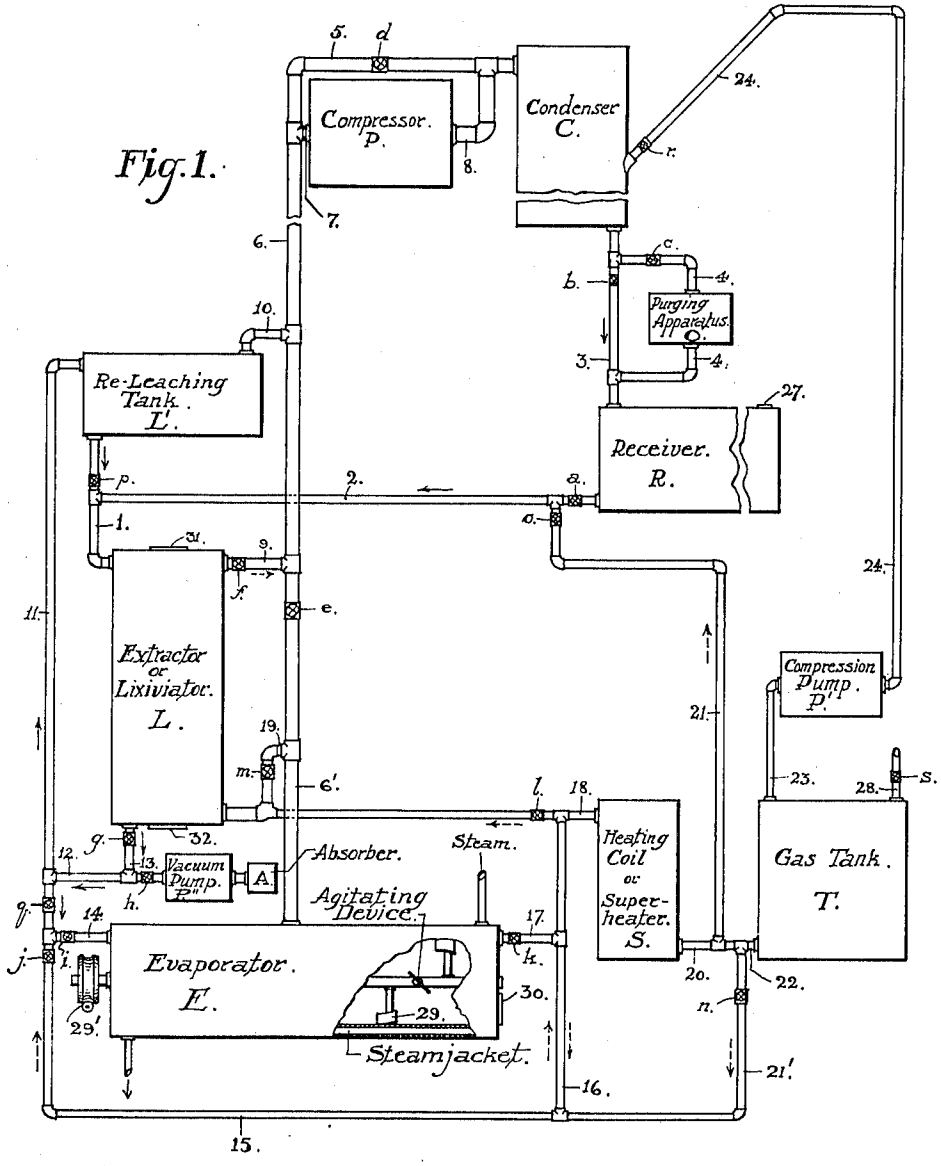
APPLICATION FILED DEC. 2, 1918.

1,314,237.

Patented Aug. 26, 1919.

2 SHEETS—SHEET 1.

Fig. 1.



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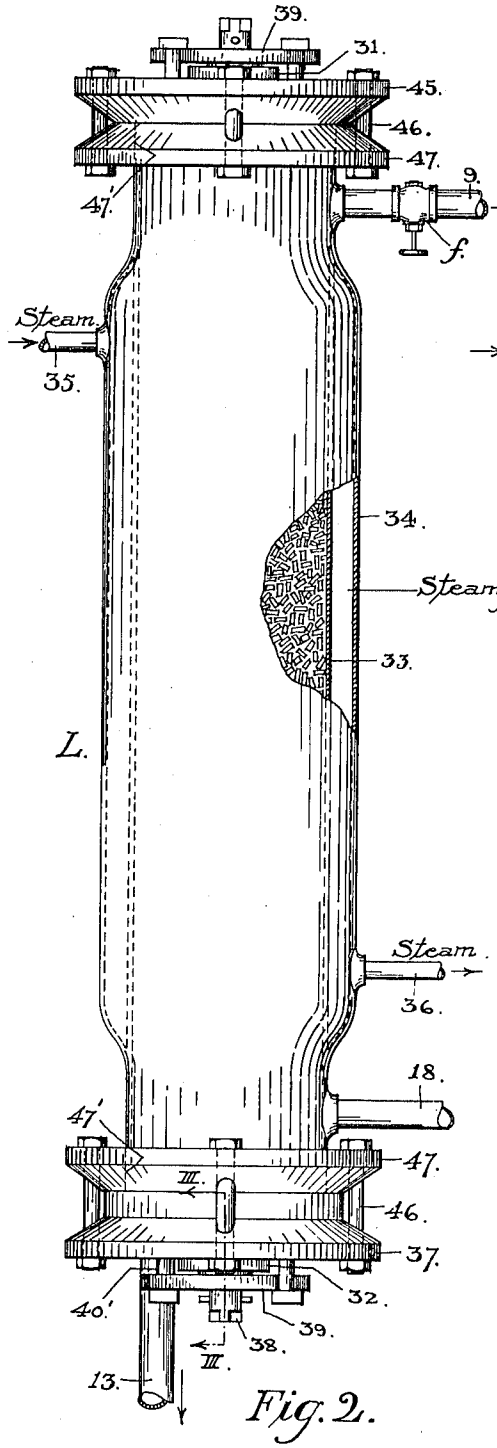


Fig. 2.

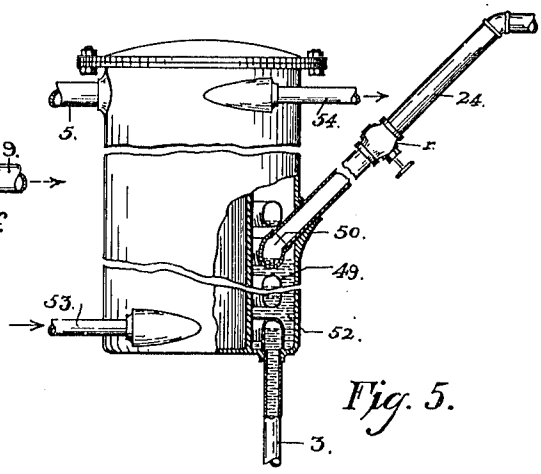


Fig. 5.

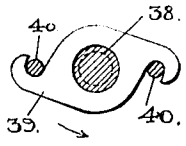


Fig. 4.

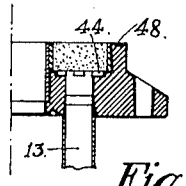


Fig. 3a.

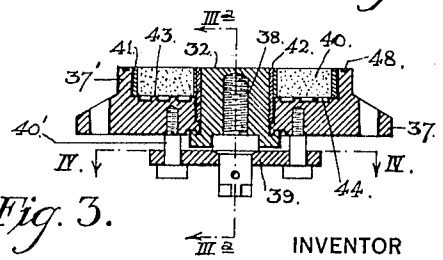


Fig. 3.

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UNITED STATES PATENT OFFICE.

EDWARD E. ARNOLD, OF GREENE, COVENTRY, RHODE ISLAND, ASSIGNOR TO NITROGEN PRODUCTS COMPANY, OF PROVIDENCE, RHODE ISLAND, A CORPORATION OF RHODE ISLAND.

PROCESS FOR RECOVERING CYANIDS AND THE LIKE FROM CYANID-BEARING MATERIAL.

1,314,237. Specification of Letters Patent. Patented Aug. 26, 1919.

Original application filed February 16, 1918, Serial No. 217,484. Divided and this application filed December 2, 1918. Serial No. 264,924.

To all whom it may concern:

Be it known that I, EDWARD E. ARNOLD, a citizen of the United States, residing at the village of Greene, town of Coventry, in the county of Kent and State of Rhode Island, have invented certain new and useful Improvements in Processes for Recovering Cyanids and the like from Cyanid-Bearing Material, of which the following is a specification.

This invention relates to an improved process for recovering alkali metal cyanid and the like from material which contains, intimately mixed with said cyanid, a material from which the cyanid is to be separated,—the invention having for one of its principal objects the effecting of economies in time and in apparatus needed for the extraction and recovery of, for example, sodium cyanid from material containing such cyanid intimately mixed with other substances, such as sodium carbonate. Another object of my invention is to provide a method for economically handling a volatilizable solvent, especially as regards its application to the material to be treated, whereby to more efficaciously dissolve the solute, and as regards speeding up or accelerating the subsequent volatilization of such solvent.

I further aim to effect economies in converting the solvent from its gaseous to its liquid phase, and in the time hitherto lost in eliminating occluded solvent from the pores or interstices of the leached or lixiviated mass, after the bulk of the solvent has been withdrawn.

These and other objects of my invention will be hereinafter referred to and the novel combination of steps constituting my improved process, will be more particularly pointed out in the claims appended hereto.

In the drawings, which form parts hereof and in which like reference characters designate like parts throughout the several views, I have exemplified a preferred apparatus in which my process may be effectuated and have described the preferred mode of effectuating said process in said apparatus; but as I am aware of various changes and modifications which may be made both in said process and apparatus, I desire to be limited only by the scope of the claims.

Referring to the drawings:

Figure 1 is a diagrammatic representation of a preferred embodiment of my lixiviating and evaporating system.

Fig. 2 is a front elevation, partly broken away, of, and more fully showing, the lixiviator which appears in the diagrammatic representation aforesaid.

Fig. 3 is a vertical detail section, taken on the line III—III of Fig. 2.

Fig. 3^a is a fragmentary detail section, taken on the line III^a—III^a of Fig. 3.

Fig. 4 is a section taken on line IV—IV of Fig. 3; and showing in plan the locking- or hook-plate for the closure or plug of the lixiviator; and

Fig. 5 is a front elevation of the condenser, the latter being partly broken away to show pipe connections.

In the U. S. patent to John E. Bucher, No. 1,120,682, dated December 15, 1914, there is disclosed a process for forming alkali metal cyanogen compounds, such as sodium cyanid; the material in which the cyanogen compound forming reaction is to be effected, being preferably, although not necessarily briqueted. This material comprises a substance capable of acting as the source of the base of the cyanogen compound to be formed, *e. g.* sodium carbonate, intimately mixed with suitable carbonaceous material, such as powdered coke, charcoal, or the like, or a mixture of such carbonaceous material.

There is also preferably present in said mass, catalytic material, such as finely divided iron or the like, and after these materials have been briqueted (or not, as the case may be), they are subjected to nitrogen gas, or to a gas such as producer gas, comprising nitrogen, and also to heat.

The temperature at which the reaction may be effected may be considerably varied, but it is preferably in the neighborhood of 1000° C.

In the hereindescribed process, the cyanized material resulting from this or a similar reaction, is preferably leached or lixiviated with a volatilizable solvent, such

as liquid ammonia, which solvent should preferably be capable of extracting or selectively removing the alkali cyanid from the other solid reaction residues.

I make no claim herein to the discovery of this preferred solvent, *per se*, and in general do not desire to be limited to the solvent specified.

I also do not desire to be limited in any way to the mode of initially producing the cyanogen compound sought, the present invention being concerned more especially with an improved method of handling a solvent of the character in question whereby, in one aspect of my invention, to expeditiously extract alkali cyanid from substances from which it otherwise is difficultly separated.

Referring now to Fig. 1, let it be assumed that the extractor or lixiviator, L, has been charged with cyanid bearing material, such as the briquets aforesaid. This receptacle is in connection through pipes 1 and 2 with a receiver R. This latter may initially be charged with the solvent in its liquid phase, and a valve *a* controls the flow of the liquid from said receiver to the extractor.

The receiver is connected with a suitable condenser C, by means of a pipe 3 in which is a valve *b*, and a by-pass 4 containing suitable purging apparatus O, hereinafter referred to, connects the sections of pipe 3 disposed upon opposite sides of valve *b*. It it also preferable to provide a valve *c* in the by-pass 4. The condenser has connected thereto a pipe 5, preferably of somewhat larger size, in which pipe there is also preferably disposed a valve *d;* the pipe 5 in turn being connected to a riser or pipe 6. A suitable pump or compresser P, may have one end thereof connected as at 7 with the riser aforesaid, while a connection or pipe 8 at the other end of said compresser connects with the pipe 5 adjacent to the condenser.

Pipe 6, shown in Fig. 1 as an extended riser, although it obviously may be otherwise disposed, preferably has therein a valve *e*, and for convenience of description the section of the riser below valve *e* has been designated 6′.

In the figure it will be observed that a connection 9 is provided between the upper end of the extractor and the lower end of the pipe 6, and in this connection there is located a valve *f*. The pipe 1, previously referred to, may conveniently be extended up to the bottom of the releaching tank L′, and, in order to vent the gas from this tank when the latter is being filled, a connection 10 is provided between the upper side of said tank and the riser 6.

Extending down from the opposite side of the tank L′, is a pipe 11 from which laterally extends a pipe 12 at the end of which is located a vacuum pump P″. A pipe 13 connects the lower end of the lixiviator with pipe 12; pipe 13 having therein a valve *g*, and the pipe 12 having a valve *h* therein, between pump P″ and pipe 13.

Immediately below the connection of the pipe 14 with pipe 11, and in the latter, I have shown a valve *j*, and the lower end of pipe 11 is in connection with a pipe section designated 15. From this latter extends up a pipe 16, which is disposed at the right hand end of the evaporator, as shown in Fig. 1; pipe 16 being connected to said end of the evaporator via pipe 17.

A valve *k* is located in pipe 17, and the upper end of the pipe 16 is in connection with a pipe 18 which extends between the lixiviator and a heating coil or super-heater S. To the left of the connection of pipe 16 with pipe 18 is valve *l*, and the left hand end of pipe 18, adjacent the lixiviator, has connected thereto a pipe or conduit 19, which in turn connects with the riser section 6′. In connection 19 there is preferably located a valve *m*. The super-heater S is connected at its lower extremity by a pipe 20 with a pipe 21—21′, which extends up from the right hand end of pipe 15 to the pipe 2 aforesaid, as shown. A connection 22 opposite the pipe 20 places pipe 21 in connection with a gas tank or reservoir T, and the latter is in turn connected with a compression pump P′, by a pipe 23.

The compression pump is in connection with a part of the condenser, as shown in Fig. 5; a pipe 24 being provided for this purpose. In the upper end of the pipe 21′ I preferably locate a valve *n*, which, as hereinafter described, controls the flow of gas from the tank T direct to the evaporator. A valve *o*, preferably at the upper end of pipe 21, similarly controls the flow of said gas into the pipe 2.

I also prefer to provide a valve *p* in pipe 1 above the connection of pipe 2 with the latter and a valve *q* in the pipe 11 leading down from the re-leaching tank, below the connection of pipe 12 with said pipe 11. In pipe 24 is a valve *r* which may be closed when the purging apparatus O is in use, on account of the backing up of liquid in the condenser coils at such time.

Ammonia may be introduced into the system, either in liquid form directly into the receiver, by removing the plug 27 of the latter,—or, in gaseous form through the same pipe 28, through which nitrogen may be supplied; valve *s* controlling the flow of gas through this pipe.

The apparatus also preferably involves the provision of a suitable agitating or stirring device 29 in the evaporator; this device being preferably driven by a worm and wheel drive 29′ whereby to both facilitate

the complete volatilization of solvent from the cyanid crystallizing out in the evaporator,—when the steam jacket of the latter, or its equivalent, is in operation,—and, also in manner hereinafter referred to, to favor the formation of fine crystals of said cyanid. Indeed, if the apparatus be not provided with an agitating device, the cyanid, when operating on a large scale, will frequently solidify into a massive block or cake which can only be removed or handled with difficulty.

When the product is in the form of fine crystals or relatively small masses, it can be removed from the evaporator through a suitable opening; the latter normally being plugged as at 30.

The openings through which the preferably briqueted charge is introduced into and discharged from the lixiviator are correspondingly plugged as at 31 and 32.

A few details of the apparatus will now be considered before discussing the operation of the system as a whole.

The lixiviator, as shown in some detail in Fig. 2, preferably comprises a cylindrical container 33, which is provided with a steam jacket 34; live steam being introduced into the latter, as desired, through a pipe 35, and the exhaust steam passing out by way of a pipe 36.

It may be here noted that all of the various receptacles, their plugs or closures, and connections should be adapted to withstand reasonably high pressures; pressures of 150 pounds to the inch, or more, being frequently attained in the lixiviator and other parts of the system. Thus, the plug 32 above referred to, is preferably, as shown in detail in Fig. 3, received into a close fitting aperture in the head 37 of the lixiviator, suitable provisions being made, as shown, for insuring a gas-tight closure; the plug being retained firmly in position by backing out its screw 38 against a hook- or retaining-plate 39, to place the bolts 40′ of the latter under tension.

When the screw 38 is reversely turned, or slackened, plate 39 may be rotated arrowwise, as viewed in Fig. 4, to permit the plug 32 and its screw to be withdrawn.

The head 37 is provided with a filter block 40 through which the cyanid containing solvent passes, to free it from any pulverulent material in the charge being leached; and, for convenience, this block may be secured in position by pouring molten lead or the like therearound, as at 41; a sleeve 42 being driven into a suitable recess in the head to provide an inner wall for the retaining material.

The filter block may be made of any suitable material, and rests upon annular ribs 43, which project upwardly from the bottom of the recess in the head in which said block is located, so as to provide below the filter block a series of channels 44. As shown in Fig. 3ª, the channels 44 are in connection with each other and with the pipe 13, which may be tapped into the head.

At the upper end of the lixiviator is a head 45, similar to that just described, except that it is not provided with the cylindrical extension 37′, since, obviously, no filter block is needed therein.

The heads 37 and 45 are held by bolts 46, firmly in engagement with annular flanges 47, secured as at 47′ to the respective ends of the casing 33; and gas-tight connections are made between these rings and their heads by suitable gaskets or seals 48.

It may be well at this point to consider a further detail, before discussing the mode of operation of the apparatus, and the steps of the process which may be carried out by means of such apparatus.

As shown in Fig. 5 the condenser C may comprise a coil 49, preferably although not necessarily of somewhat flattened tubing to allow the condensed ammonia to flow down therethrough, while leaving a space 50 thereabove in the tube, to permit of gases in this space being drawn off through the pipe 24, as shown. Suitable refrigerating fluid 52 may be circulated through the condenser as by means of pipes 53 and 54 and the liquid ammonia passes off from the condenser through the pipe as above described.

The preferred mode of effectuating the process in the system given by way of exemplification, is as follows:

Assuming that the lixiviator is cold and has been charged with alkali metal cyanid bearing material; that the receiver contains the solvent to be employed, in liquid form, *e. g.*, liquid ammonia; and that the various valves in the system have all been closed with the exception of valves *g*, *h*, *q* and *i*.

The vacuum pump P″ is now started, to exhaust the air from the lixiviator, evaporator and, if the releaching tank be provided, as is to be preferred, it also, together with the various connections thereto, will be correspondingly exhausted.

If desired, in order to make the removal of atmospheric oxygen more complete, the valve *o* may be opened, admitting compressed nitrogen gas, or mixed nitrogen and gaseous ammonia, from the gas tank T, which passes up through the pipe 21, to the pipe 2, and thence into the releaching tank, extractor and evaporator. Valve *o* may then be closed and the nitrogen removed from these several receptacles by the vacuum pump; any gaseous ammonia present in this nitrogen being absorbed in the absorber or scrubber A.

Valves *g*, *h*, *q* and *i* are now closed and valve *a* is opened to admit liquid ammonia, via pipes 2 and 1, to the lixiviator. After

the latter has been filled, valve *a* is closed, and valves *g* and *o* may be opened, so that compressed gas flows into the top of the lixiviator and drives the liquid ammonia through pipes 13 and 12, and up through pipe 11, to the releaching tank.

Valves *g* and *o* are now closed and valve *p* is opened to permit the solvent (partially saturated with alkali cyanid) in tank L′, to flow down by gravity, into the lixiviator, whereby to more nearly complete the removal of alkali metal cyanid, cyanamid, or the like from the lixiviator. Valves *f* and *d* may be cracked to relieve the gas pressure in the lixiviator, if this tends to prevent a rapid discharge of the liquid from the tank L′.

The above circulating operation may, if desired, be repeated several times in order to more nearly saturate the solvent. If the charge of material in the extractor is particularly rich in cyanid, it may be necessary to supply additional liquid ammonia from the receiver,—the saturated solution, first being drawn off into the evaporator, by opening the valves *g*, *q* and *i*.

Should sufficient ammonia evaporate at any time to prevent the proper transfer of the solvent up through the pipe 11 to the releaching tank, in the manner described, the latter may be exhausted, by operating the compressor P, or even the smaller compressor P′, for a short time.

Assuming now that the evaporator has been properly charged with quite well saturated solution; the problem then arises as how best to speedily eliminate the liquid ammonia, or the like, occluded in the pores and interstices of the mass in the lixiviator. This occluded liquid is considerable in quantity and evaporates but slowly, even though steam be turned into the jacket of the lixiviator, on account of the poor heat-conductive qualities of the mass in the latter. It has hitherto, therefore, proven to be a serious problem how to shorten the time required for the removal of this occluded liquid, and the solution of this problem is one of the objects of the present invention.

Another object of the invention concerns the proper and yet reasonably rapid evaporation of liquid in the evaporator. On account of the fact that the mass being treated, at least initially, in this receptacle, is wholly liquid, it would not except for the difficulty hereinbelow pointed out, involve a serious problem to speedily evaporate such liquid by means of steam supplied to the evaporator steam jacket. The difficulty is, however, that this heat is largely supplied to the bottom and sides of the liquid mass, which is of course a readily volatilizable one, and the vapor formed rises in the form of bubbles to the surface of the liquid, where it bursts through, entraining the cyanid or the like, and carrying the latter along through the conduit through which the vapor escapes, until it causes considerable trouble in the piping and especially at the valves, by reason of its accumulation at such points. It is for this reason that I prefer to provide an extended riser, such as that designated 6′, through which the generated gas may escape. This difficulty may, however, be further and more nearly completely overcome by not starting the steam jacket of the evaporator into operation until much of the liquid in said evaporator has been driven off by the passage of warm gas over the liquid surface.

This gas may be any suitable one which will not injure the product or solvent, and the inert gas nitrogen, being entirely innocuous, lends itself readily to the purpose.

I do not however desire to be limited to this, since various gases may be employed, or even mixtures of gases.

Thus, as ammonia gas is evolved in the evaporator, it, obviously, is available for use as a heat medium; and indeed, as will be pointed out hereinafter, when, for example. nitrogen gas is thus used in the system, it becomes intermingled with the gas evolved upon the evaporation of the solvent.

Referring again to Fig. 1, therefore, the superheater S, as it may for convenience be termed, is started into operation, and, all other valves being closed, the below mentioned valves will be opened.

Gas will pass from the tank T through the connections 22 and 20 to the superheater and thence via pipes 18 and 16 to pipe 17, and also to pipe 15, the lower end of pipe 11, and connection 14, to the respective ends of the evaporator, the valves *k*, *j* and *i* being open at such time.

By proper adjustment of valves *j*, *i* and *k*, the flow of warm gas into the opposite ends of the evaporator, may be adjusted, so that a substantially equal flow may be obtained; said gas, together with the vaporous ammonia or the like, given off at the surface of the body of liquid in the evaporator, passing up through the riser 6′, and as it is usually preferred to direct this warm gas through the lixiviator to hasten the evaporation of occluded liquid therein, the valve *e* may be closed, or nearly closed, as desired, and the valves *m* and *f* are opened. The expression "warm gas," above mentioned, is intended to be used as a relative term which contemplates that the temperature of the gas in question is rather materially above that at which the solvent copiously volatilizes.

Normally during the just described operation, the valve *d* may be open, and, the compressor being idle, this will prevent the flow of gas through connections 7 and 8. However, the pressure of gas generated by the

evaporation of liquid from the receptacles L and E, will normally be sufficient to enable the condensation of said gas in the condenser; whence the liquid solvent, now free from cyanid, may pass directly down into the receptacle R.

By the procedure above set forth, not only is the volatilization of liquid in the evaporator hastened, but there is a markedly less tendency for the carrying over or up of cyanid. Also the evaporation of the occluded liquid solvent in the lixiviator is greatly expedited.

If at any time the rate of volatilization in the evaporator becomes excessive, either the heat imparted by the superheater may be reduced, or the valve *n* may be opened and relatively cool gas admitted to the streams of warm gas entering the evaporator; this cool gas passing down through pipe 21′ and entering pipes 15 and 16.

When the liquid in the receptacle L has been completely removed, as by this procedure it will be in a comparatively short time, the valve *e* may be opened and valves *f* and *m* closed, so that the gas leaving the evaporator will pass directly up through the riser section 6 without entering the lixiviator.

If for any reason, as on account of the nature of the charge introduced into the lixiviator, difficulty should be encountered in removing the occluded liquid from said charge within a reasonable time, evaporation thereof can be speeded up by opening valve *l*, and closing the valve *m*, whereby to admit warmer gas directly to the lixiviator; this, together with the heat at such time supplied by means of the steam jacket of the lixiviator, very speedily evaporating the residual occluded liquid.

After evaporation has been so far reduced that the pressure generated in the condenser is not sufficient to effectively enable this apparatus to perform its function, the valve *d*, if desired, may be closed and the compressor P started. This exhausts the gases from the extractor and evaporator, and as it will frequently be necessary to gain access to the former sooner than the latter, the valves *m*, *e* and *q* (and if desired *p* and *g*) may be closed for a short time, to enable the compressor to exert its full effect upon the gas in the lixiviator, to produce a reasonably high vacuum therein.

After this has been accomplished the valve *f* may be closed and the valve *e* opened to allow the compressor to act wholly upon the evaporator, in like fashion.

When all of the occluded liquid in the lixiviator has been removed from the charge therein, all valved connections thereto should be closed, and the plug 32 in the head 37, may be withdrawn, to remove the charge. At such time the said charge, if the operation has been properly conducted, will be perfectly dry, and have at most therein but substantially a trace of cyanid.

The gas present in the space 50, above the condensed fluid flowing down through the condenser, may be tapped off continuously or intermittently as desired through the pipe 24, through the operation of the compression pump P′ which delivers said gas under pressure to the gas tank T.

Thus, if nitrogen be initially supplied from tank T to the system, this nitrogen will be returned to said tank, and unless some special apparatus be provided for removing the ammonia gas mixed therewith, some of this latter gas also will pass through the pump P′ into the gas tank. Under a system such as I have described however there is no objection to this, and in so far as the system is concerned, it may be operated substantially as well with ammonia gas alone.

On account of the fact however that, as above noted, such a relatively inexpensive gas as nitrogen, permits of its use for the purpose of sweeping out traces of atmospheric oxygen or the like, and as it is not only cheaper than ammonia, but also equally harmless to the process, I prefer to use it, in manner aforesaid. After the system has been in operation for some time, the gas thus used for sweeping out purposes, will comprise a mixture of nitrogen and ammonia, and to prevent loss of this latter, the absorber A is connected to the delivery end of the vacuum pump P″.

As various impurities are, during the course of an extended operation of apparatus of this description, apt to accumulate in the solvent employed, it is desirable to provide means to remove such impurities from time to time. To this end, the purging apparatus O is disposed in the by-pass 4, so that the liquid passing from the condenser to the receiver, instead of passing directly to the latter through pipe 3, may, by the closing of valve *b* and opening of valve *c*, be forced to pass through said purging apparatus. At such time, with the arrangement shown, it is preferable not to operate the pump P′, on account of the rise in level of liquid within condenser coil.

I particularly desire to emphasize the importance of using a suitable gaseous medium as a means for conveying heat to the interior of the mass in the lixiviator, as a means for effectively removing occluded liquid therefrom. The importance of thus speeding up this phase of the operation will be appreciated upon consideration of the fact that by thus very materially cutting down the time required for the removal of this liquid from said mass, the apparatus can in any given period of time handle the

output of a very much larger cyanizing unit; so that a marked economy is effected, not only in time, but in the cost of installation, space occupied by the plant, and other costly items.

In like fashion, the speeding up of the rate of evaporation of liquid in the evaporator through the practical overcoming of the difficulty above noted, similarly effects worth-while economies of operation.

I also desire to direct attention to the utilization, in the preferred mode of effectuating my process, of the still relatively warm gases passing off from the evaporator, as a means for hastening the volatilization of occluded liquid in the lixiviator.

The system as a whole, moreover, is a very flexible one and adapts itself to varying conditions; and while all of the features shown therein are by no means essential thereto, in so far as the broadest aspects of my invention are concerned, they nevertheless add to the efficiency of the system as a whole. Thus, while I am aware that it is not necessary to provide a releaching tank in order to with ease nearly completely saturate the solvent with its solute; still the combination of this tank with the lixiviator and associated parts, permits of the solvent being exceedingly well circulated or flushed through the charge to be leached, and this without loss of the solvent, and with very appreciable gain in recovering cyanid from all parts of the leached mass. The importance of this will be seen when it is pointed out that if one merely allows liquid ammonia, for example, to run into the lixiviator, and even though it be allowed to stand in this receptacle for 20 or 30 minutes, when it has been drawn off, and the briquets or the like are analyzed to ascertain the cyanid content remaining therein, it will be found usually that there is a very much larger percentage of cyanid in the briquets at the top of the charge than in those in the center; and that for some reason, the briquets in the lower part of the charge are richer in cyanid than those in the center, although not normally so rich as those at the top.

The combination of the jets or currents of warm gas over the surface of the liquid in the evaporator, with the means for applying heat superficially to this receptacle, *e. g.*, the steam jacket, is also of importance, in that a large part of the liquid may be first removed, working from the surface of the liquid down, so to speak, and after much of this liquid has thus been evaporated, the bringing into play of the steam jacket or the like, accelerates the last portion of this operation, which otherwise is quite slow

The effect of the superficial heating of the charge in the evaporator, when much of the liquid in the latter has been driven off and the mass is approaching its crystalline condition, has an important bearing upon the ultimate condition of the recovered cyanid; since at such time, the steam jacket is preferably in operation and the agitating apparatus is being driven in a direction to churn or thoroughly agitate the mass, but on account of the refrigerating effect of the volatilizing solvent, there is a tendency for this mass, especially as it becomes thick with crystals, to cake or solidify in portions thereof and thereby to resist the action of the agitator. This calls for expenditure of more work in driving the latter, and the product is apt not to be as finely divided as might be desired. On the other hand the flow of warm gas over the surface of the charge at such time, supplements the heat imparted to the charge by the walls of the receptacle and tends to maintain the temperature of the charge as a whole, more nearly uniform; with the result that the product is improved and the expenditure of energy in turning the agitating paddles is reduced.

Finally, the evaporation of solvent in the above described manner, coupled with the utilization of the pressure so obtained to enable the condensation of gas or vapor in the condenser, and this especially for that part of the time during which the condenser is idle, results in great fuel economy, for the following reason: With this method the energy for forcing the ammonia from the evaporator to the condenser coils, and for producing the pressure at which the ammonia will condense in the coils, has its source in the superheater and in the heat of the steam in the steam jacket or jackets, and all of this heat is used to produce the desired results.

As above noted, when substantially all of the liquid ammonia is vaporized, the evaporator is left full of gaseous ammonia, which must be withdrawn and since it must be delivered to the condenser coils under high pressure, a compressor is required; but in this case it need be but a relatively small one. Indeed, it is even possible to omit the compressor P, relatively small though it is, and rely upon the pump P′ to remove this residual gas; it being understood, however, that in such case this remaining gas will not be condensed but will be delivered to the tank T under pressure, ready for return to the system through the superheater, or otherwise, as desired.

If the gas delivered to the tank T be not allowed to cool, it will normally be quite warm, in most cases, after being compressed.

The above described system and mode of operating it, has the further advantage that if liquid ammonia be the solvent used, as is to be preferred,—only anhydrous ammonia need be used as is to be desired where substantially pure cyanid is the product sought.

In conclusion, I desire to point out that the present case, which is a division of my application Serial No. 217,484, filed Feb. 16, 1918, concerns more particularly the process described in said parent application, the latter having been restricted in the claims thereof, to the preferred apparatus or system through the intermediacy of which said process may be conveniently effectuated.

Having thus described my invention, what I claim is:

1. The process of recovering alkali metal cyanid from a porous mass containing said cyanid intimately mixed with material from which said cyanid is to be separated, which comprises, leaching said mass with a selectively acting solvent capable of extracting said cyanid from said mass, separating the bulk of said solvent from said mass, evaporating the separated solvent by the application of heat thereto, and passing a portion at least of the evaporated solvent, while at a temperature above that at which said solvent copiously volatilizes, through the pores of the leached mass to volatilize the residual solvent occluded in said pores.

2. The process of recovering alkali metal cyanid from a porous mass containing said cyanid intimately mixed with material from which said cyanid is to be separated, which comprises leaching said mass with a selectively acting solvent capable of extracting said cyanid from said mass, separating the bulk of said solvent from said mass, and passing a gas the temperature of which is materially above that at which said solvent copiously volatilizes, through the pores of the leached mass to volatilize the residual solvent occluded in said pores.

3. The process of recovering alkali metal cyanid from a porous mass containing said cyanid intimately mixed with material from which said cyanid is to be separated, which comprises leaching said mass with a selectively acting solvent capable of extracting said cyanid from said mass, separating said solvent from said mass, and evaporating the solvent by directing over and in contact with its surface a current of gas under pressure and at a temperature materially above that at which said solvent boils, to freely volatilize said solvent while maintaining substantially at a minimum the quantity of cyanid entrained with the gas being liberated from said surface.

4. The process of recovering alkali metal cyanid from a porous mass containing said cyanid intimately mixed with material from which it is to be separated, which comprises subjecting said mass to the action of a selectively acting solvent capable of extracting said cyanid from said mass, expelling the bulk of said solvent from said mass by gaseous pressure, elevating said expelled solvent by said pressure to a level above that of the mass aforesaid, and flowing said solvent back into said mass by gravity, whereby to again subject the residual cyanid in the pores of said mass to said solvent to remove cyanid from all parts of the mass being treated to substantially the same extent throughout.

5. The process of recovering alkali metal cyanid from a porous mass containing said cyanid intimately mixed with sodium carbonate, which comprises, treating said mass with liquid ammonia, withdrawing the so formed solution of said cyanid, recovering the residual ammonia from the pores of said mass by passing therethrough a warm gas, composed in part at least of ammonia, heating the withdrawn solution to volatilize off the ammonia therefrom, and subjecting the gas passing off from said mass and from said withdrawn solution to the action of a cooling medium while utilizing the pressure of said gas, resulting from the volatilization thereof by heat, to convert gaseous ammonia present therein to the liquid phase.

6. The process of treating alkali metal cyanid bearing material to obtain said cyanid therefrom, which comprises, introducing said material into an air-tight receptacle, exhausting the air from the interstices and pores of said material, admitting an innocuous gas to said receptacle and thereafter removing said gas, to, in effect, sweep out remaining traces of oxygen, repeatedly flushing said material with liquid ammonia, withdrawing said ammonia, volatilizing it to leave the dissolved cyanid in crystalline form, and recovering the ammonia occluded in the pores of said material by passing therethrough warm gas, consisting in part at least of said volatilized ammonia.

7. The process of treating alkali metal cyanid bearing material to obtain said cyanid therefrom, which comprises, introducing said material into an air-tight receptacle, flushing said material with liquid ammonia, withdrawing said ammonia, volatilizing it to leave the dissolved cyanid in crystalline form, and recovering the ammonia occluded in the pores of said material by passing therethrough a heat supplying medium consisting in part at least of ammonia in the gaseous phase.

8. The process of producing substantially pure cyanid which comprises forming an alkali metal cyanid in a porous mass which contains alkali metal carbonate and carbon, treating said mass to separate said cyanid therefrom, in part at least through the intermediacy of an extracting solvent the boiling point of which is materially below that of water, heating the cyanid containing solvent to convert said cyanid to crystalline form, and recovering the residual solvent occluded in the pores of said mass, by introducing into said pores a gas at a tempera-

ture materially above that at which said solvent volatilizes.

9. The process for recovering alkali cyanid from material bearing the same, which comprises dissolving the cyanid, present in said material, in a selective extracting agent, withdrawing the bulk of the so formed solution from the undissolved residues of the first operation, imparting heat to said solution to volatilize said agent, and conducting a part at least of the relatively warm gas, so formed, through said residues to volatilize the occluded solvent in the pores of the latter.

10. A process for recovering alkali metal cyanid from porous material bearing the same, which comprises lixiviating said material with a selective solvent for said cyanid, withdrawing the so formed solution from the undissolved residues, heating said withdrawn solution to volatilize said solvent, heating said residues to facilitate the evaporation of any residual and occluded solvent therein, and treating said residues with the selective solvent in vaporous condition to complete the evaporation of said residual solvent, said selective solvent, when thus used in vaporous condition, being at a temperature materially above the boiling point of said solvent.

In testimony whereof I have affixed my signature, in the presence of two witnesses.

EDWARD E. ARNOLD.

Witnesses:

HELEN M. BARNBROOK,

HOWARD C. RIPLEY.