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N. W. F. PHILLIPS ETAL

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METHOD AND APPARATUS FOR CONVERTER RESIDUE DISCHARGE

Filed Feb. 3, 1965

2 Sheets-Sheet 1

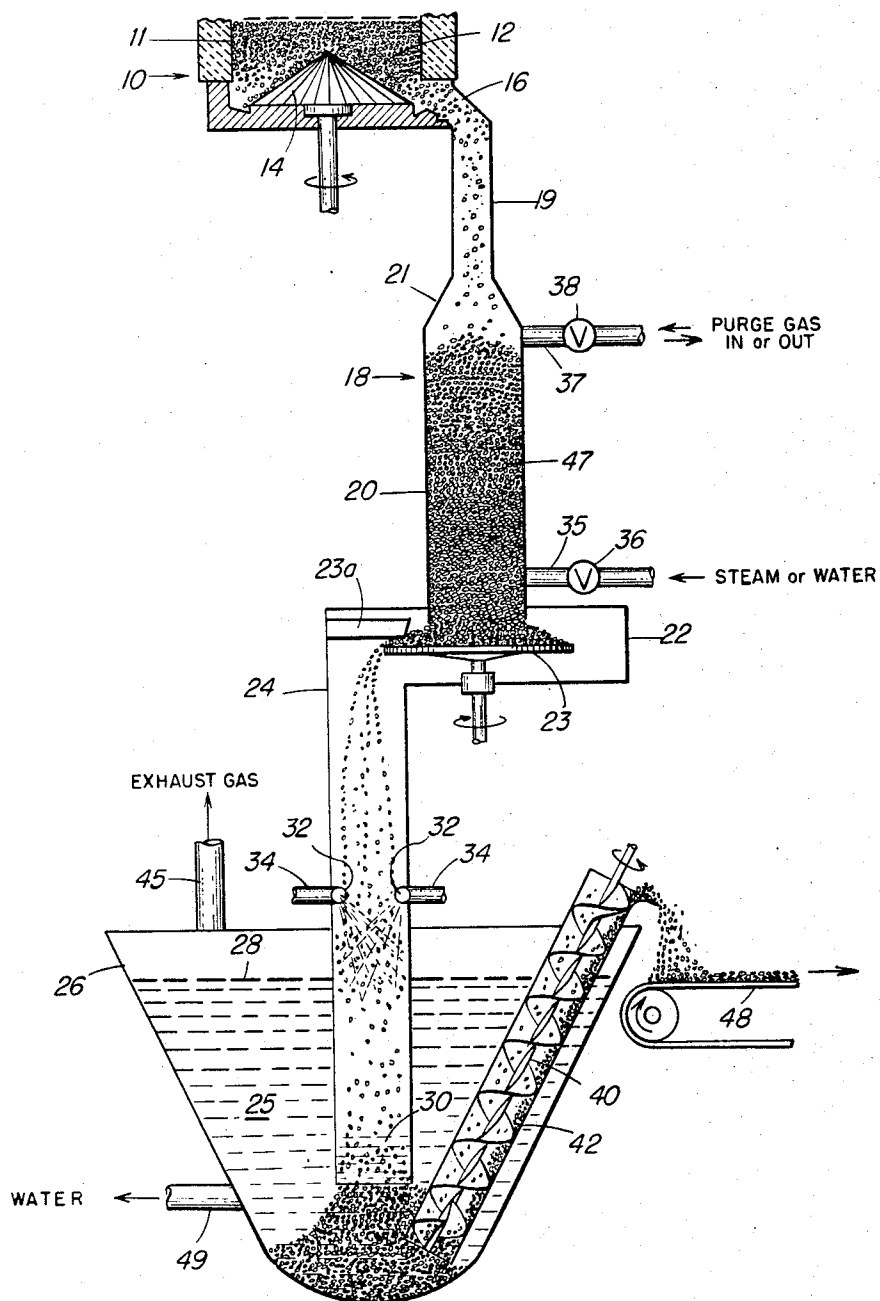


FIG. 1

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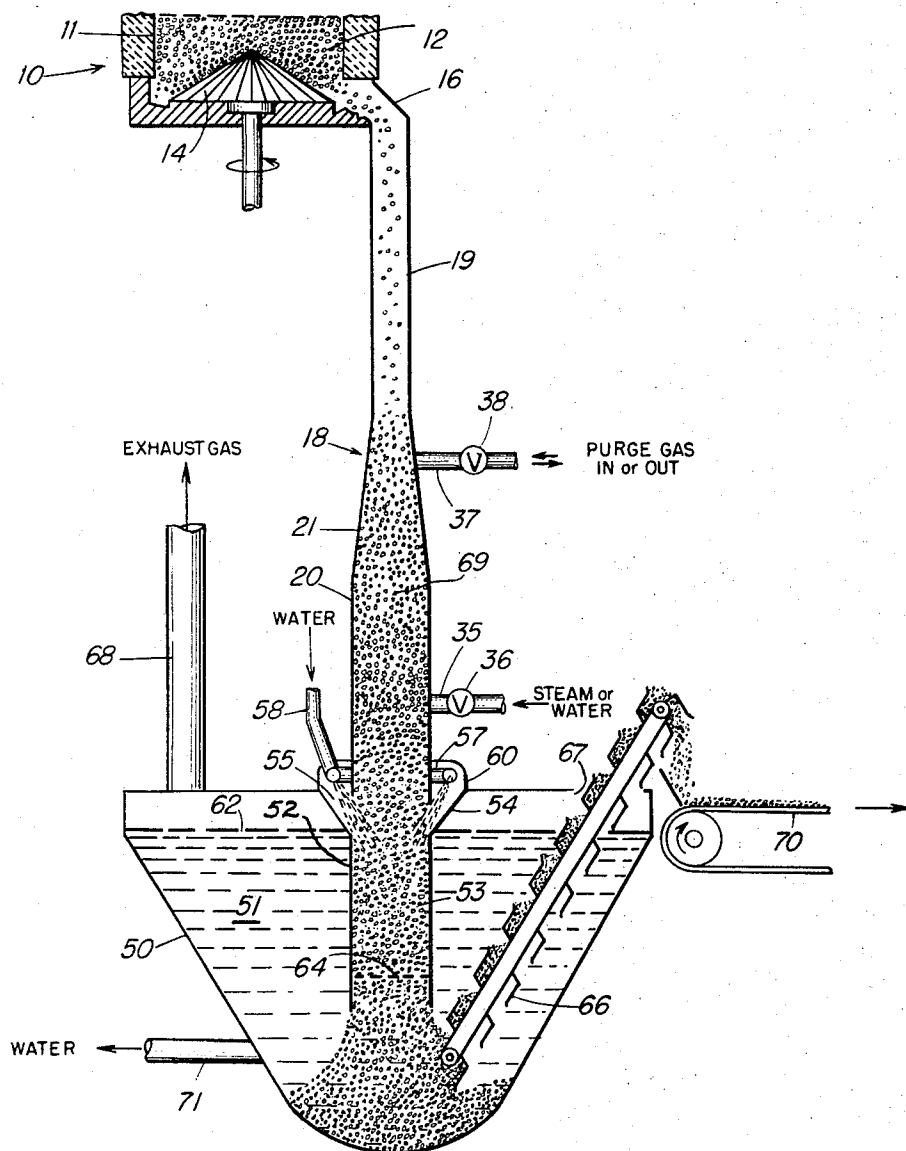


FIG. 2

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METHOD AND APPARATUS FOR CONVERTER RESIDUE DISCHARGE

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14 Claims. (Cl. 75-68)

This invention relates to the so-called subhalide distillation of aluminum from alloys or like metallic materials containing aluminum together with other metals. In particular, the invention relates to methods for discharging, from an aluminum subhalide distillation system, materials constituting the solid residue of such alloys which remains after extraction of aluminum therefrom in the system.

The production of purified aluminum metal from crude aluminum-containing alloys by subhalide (e.g. monochloride) distillation, as disclosed for example in United States Patent No. 2,937,082, involves exposure of the alloy to normal aluminum halide (e.g. aluminum trichloride, $AlCl_3$) in gaseous state, at an elevated temperature. The aluminum in the alloy reacts with the halide gas to form the corresponding subhalide of aluminum, as a gas, which is withdrawn and subjected to further treatment to yield purified aluminum metal. Thus, by way of example, the crude alloy obtained by direct reduction of bauxite or other aluminum ore, containing aluminum, iron, and other elements, may be heated, e.g. to a temperature ranging upwardly from about 1000° C., and exposed to aluminum trichloride gas whereby the aluminum in the alloy reacts with the gas to form aluminum monochloride gas (i.e. $AlCl$).

The foregoing operation, in accordance with presently preferred practice, is performed in a suitable converter or furnace having an upright confined chamber to which the alloy is charged in granular form, viz. as fine particles, granules or lumps ranging e.g. up to 3 inches in size, to provide a granular mass of alloy substantially filling the chamber. This mass, heated to the aforementioned temperature as by electric current passed therethrough between suitably disposed electrodes in the converter, is exposed to a continuous flow of pre-heated aluminum trichloride gas which passes upwardly through the alloy mass in the chamber from a gas inlet near the lower end of the chamber. The aluminum monochloride gas produced in the chamber, together with the unreacted portion of the introduced trichloride gas, is withdrawn through a gas outlet located near the upper end of the chamber for treatment to decompose the monochloride gas to yield aluminum trichloride gas and the desired purified product aluminum metal.

In continuous operation of the converter, successive quantities of fresh (i.e. unreacted) granular alloy are supplied to the top of the alloy mass in the chamber and successive quantities of the alloy are correspondingly withdrawn from the lower end of the mass, so that the introduced alloy moves downwardly through the chamber while being progressively depleted of aluminum by reaction with the trichloride gas flow therein. The alloy at the lower end of the mass is thus a spent residue, i.e. substantially exhausted of aluminum, and has a composition typically in the following range of proportions:

	Percent
Iron	50-70
Aluminum	3-15
Silicon	5-15
Titanium	3-8
Carbon	7-12

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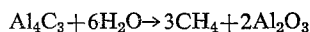
Withdrawal of this residue from the converter chamber is conveniently accomplished by suitable mechanical means such as a rotary extractor cone positioned at the floor of the converter chamber to engage the lower end of the alloy mass and arranged to expel the residue, in the form of lumps and granules, through one or more residue outlet openings in the wall of the converter. For the sake of convenience and economy of operation it is desirable that the expelled residue be delivered to the open air for disposal; this is particularly true in the case of continuous operation of a subhalide system on any substantial scale, since such operation involves the successive expulsion of large quantities of the residue from the converter chamber of the system. In effecting such transfer of residue, however, it is necessary to prevent flow of gas between the converter chamber and the air. Gas pressure in the converter is ordinarily somewhat higher than atmospheric pressure. If the residue discharge outlet provided a path for escape of converter gas to the atmosphere, substantial losses of aluminum halide gas from the subhalide distillation system would occur, a consequence obviously undesirable from the standpoint of economy; moreover, air might enter the converter chamber, where it would interfere with the desired converter operation.

To prevent such gas flow it has been proposed to pass the residue expelled from the converter through a succession of purged hoppers separated by gas-tight valves. However, it is found that the severe conditions (including high temperature, abrasive dust and frequent operation) to which the valves are subjected tend to produce relatively rapid deterioration of the sealing surfaces of the valves.

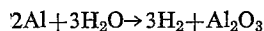
It is accordingly an object of the present invention to provide a method of discharging residue alloy from a subhalide converter, which prevents loss of aluminum halide gas from the converter in a manner eliminating the necessity for passing the residue through valves or other mechanical sealing means. A further object is to provide procedures for effecting transfer of residue alloy from a subhalide converter to the air and concomitantly water-cooling the residue to facilitate handling thereof, in a manner which affords assured prevention of gas communication between the converter chamber and the ambient atmosphere and which also prevents contact between aluminum halide gas from the converter chamber and the water employed in cooling the residue, without necessitating passage of the residue through valves or other mechanical sealing means.

To these and other ends, the process of the present invention in a broad sense contemplates discharging the residue alloy through the converter residue outlet opening or openings into a laterally confined passage e.g. extending downwardly therefrom, and advancing the residue through the passage while exposing the residue to water in liquid and/or vapor state at a locality in the passage spaced from the converter region, to effect generation of gas (by reaction of the water with water-active constituents of the residue) and thereby to provide an atmosphere or flow of such gas which extends through the passage to encounter aluminum halide gas entering the passage from the converter and acts as a purge to oppose flow of the halide gas through the passage.

In this connection it may be explained that residue alloy as discharged from a subhalide converter contains aluminum combined with carbon as aluminum carbide, which reacts with water to form methane:



The residue also ordinarily contains some uncombined aluminum, which reacts with water to form hydrogen:



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Thus exposure of the residue to water or water vapor generates gas, of which the major constituent is methane but which also includes some hydrogen. These gases are suitable for the purge atmosphere in the passage, since they are chemically inert with respect to the aluminum halide gas and the aluminum of the alloy in the converter. The amount of such gases produced in the passage in the present process is dependent, *inter alia*, on the amount of water or water vapor supplied to the residue, the proportion of aluminum (both free and combined) in the residue, the duration of water-residue contact, and the temperature at which such contact occurs.

Further in accordance with the invention, the lumps and granules of residue alloy descending through the passage are advanced through an intermediate or lower portion of the passage (herein termed the water vapor reacting zone) at a rate controlled to maintain such portion filled with a column of the residue to a predetermined level therein; and the water and/or water vapor are supplied to the residue in the passage in or below this water vapor reacting zone, so that water vapor rising in the passage from the locality of water (or water vapor) introduction passes through the column of residue together with the produced purge gas. The residue column, it may be explained, is freely permeable to gas, and hence the purge gas produced in or below the column extends through the column to the passage portion above the aforementioned predetermined level, where it encounters aluminum halide gas from the converter. Such retention of the residue as a column in the water vapor reacting zone ensures substantially complete reaction of the water vapor rising therethrough, by providing a relatively long vapor-residue contact time with thorough exposure of the vapor to the residue. Also, the residue in the water vapor reacting zone is still at a comparatively high temperature and this temperature condition further contributes to completeness of reaction of water vapor in the column.

With such operation, a purge atmosphere comprising methane together with some hydrogen is provided (i.e. by water-residue reaction as already described) extending through and above the residue column in the passage, and in the passage portion above the water vapor reacting zone this purge atmosphere is essentially free of water vapor. Such freedom from moisture is very desirable, since the purge gas, as stated, encounters aluminum halide gas from the converter in the upper portion of the passage; if water vapor reached the latter passage portion it would react with the aluminum halide gas to form acid (e.g. hydrochloric acid, when the aluminum halide gas is aluminum trichloride), creating an undesirable corrosive condition in the passage.

By appropriate selection of operating conditions, the generation of purge gas in the lower portions of the passage may be made sufficient to prevent any significant downflow of aluminum halide gas from the converter beyond the upper portion of the passage (i.e. the portion above the water-vapor-reacting zone). Alternatively, if sufficient gas for this purpose is not generated in the passage, the gas produced in the passage may be augmented by introducing additional purge gas (e.g. methane and/or hydrogen) to the passage from an external source, to provide a purge atmosphere effective to block downflow of halide gas beyond such upper passage portion. In either event, the process of the invention thus provides discharge of residue alloy from the subhalide converter while preventing loss of aluminum halide gas from the converter in any progressive or continuing sense, yet without the necessity of passing the residue through valves or other mechanical sealing means that would be subject to deterioration under the conditions involved in residue discharge operation.

In an important specific aspect the invention particularly contemplates effecting transfer of residue alloy from the discharge passage to the air through a so-called water seal. For such transfer, the passage extends downwardly

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into a body of water, opening into the water at a level far enough below the water surface so that water stands in the lower end of the passage, i.e. providing a water seal at the lower end of the passage for assured prevention of gas flow into or out of this passage end. The aforementioned water vapor reacting zone is located somewhat above the level of water in the passage, and the residue alloy descends therefrom through the passage into the body of water. In a portion of the passage intermediate the water vapor reacting zone and the water level in the passage the descending residue is cooled, as by sprays of water directed into the path of the residue, so that it enters the body of water at a reduced temperature. The residue may be withdrawn from the body of water directly to the open air, for disposal as desired.

In this way, transfer of residue to the air is effected without any gas communication between the converter and the atmosphere, and, again, without the use of valves or other mechanical sealing means; the purge atmosphere in the passage prevents flow of aluminum halide gas from the converter to the water seal, and the water seal prevents gas flow between the passage and the air, thus contributing to maintenance of the requisite purge atmosphere in the passage. Prevention of access of aluminum halide gas to the water seal is very important, since if the halide gas reached the seal it would dissolve in the water (resulting in loss of halide gas from the distillation system) and would also create corrosive conditions in the seal.

In the procedures just described, the residue is exposed to water or water vapor, with resultant production of purge gas, in three zones of the passage successively traversed by the descending residue: the aforementioned water vapor reacting zone; the passage portion in which the residue is cooled, herein termed the cooling zone; and the terminal portion of the path of residue descent, extending below the water level in the passage and herein termed the water seal zone. Specifically, in the latter two zones some generation of purge gas (i.e. methane, together with hydrogen) occurs due to reaction of residue constituents with the cooling spray and the body of water. This gas is saturated with water vapor from the cooling spray and the latter body; the water vapor rises through the passage to the water vapor reacting zone where it is essentially completely reacted (producing additional methane and hydrogen) by exposure to the column of alloy therein, so that the purge atmosphere above the latter zone is essentially moisture-free as desired.

The extent of purge gas production and water vapor evolution in and below the cooling zone is dependent on conditions of operation, particularly including the rapidity and extent of cooling. In some instances, the purge gas generated in the cooling zone and water seal zone (together with that generated in the water vapor reacting zone by reaction of the water vapor rising from the cooling zone) may be sufficient to provide the requisite purge atmosphere for preventing down-flow of aluminum halide gas beyond the upper portion of the passage. However, it is presently preferred to effect very rapid and extensive cooling in the cooling zone so that relatively little vaporation occurs therein, and to generate the major portion of the purge atmosphere in the water vapor reacting zone by supplying steam or water directly to the latter zone from an external source. Close control of the generation of purge gas in response to changing conditions (such as change in aluminum content of the residue alloy, which affects purge gas production) can thereby be effected in a facile and convenient manner, i.e. by regulating the supply of steam.

Further features and advantages of the invention will be apparent from the detailed description hereinbelow set forth, together with the accompanying drawings, wherein:

FIG. 1 is a view in vertical section, largely diagrammatic, of one example of apparatus with which the process of the invention may be performed; and

FIG. 2 is a view in vertical section, also largely diagrammatic, of another example of apparatus arranged for performance of the present process.

Referring first to FIG. 1, the apparatus there shown is arranged to effect discharge of residue alloy from the converter of an aluminum subhalide distillation system. The converter, may, for example, be of a conventionally proposed type having an upright chamber in which a mass of granular aluminum-containing alloy is heated electrically, e.g. to a temperature above about 1000° C., and exposed to a flow of aluminum trichloride gas for reaction of aluminum in the alloy with the trichloride gas to produce gaseous aluminum monochloride. The trichloride gas is supplied in continuous flow to the lower part of the converter chamber; the produced monochloride gas, together with unreacted trichloride gas, is withdrawn from the upper part of the chamber for further treatment in a decomposer region to decompose the monochloride into aluminum trichloride gas and purified aluminum metal, and the trichloride gas is recirculated from the latter region to the converter chamber for re-use thereon. Fresh alloy is introduced in successive increments to the upper end of the converter chamber, and successive quantities of alloy are expelled from the lower end of the chamber, so that the alloy traverses the chamber in a downward direction as it is progressively depleted of aluminum.

The lower portion of a subhalide converter of the foregoing type is shown at 10 in FIG. 1 in fragmentary elevational sectional view, with the converter chamber 11 illustrated as filled with a granular alloy mass 12. It will be understood that the chamber 11 contains an atmosphere of aluminum trichloride gas, at a pressure typically higher than 1 atm., and that the lower end of the alloy mass (i.e. the portion shown) comprises aluminum-lean residue alloy ready for discharge. Removal of this residue may be effected by means of an upright extractor cone 14 positioned at the floor of the converter chamber to engage the lower end of the alloy mass and rotated by suitable means (not shown) so as to expel the residue through discharge aperture structure (here for simplicity shown as a single aperture 16 opening at one side of the converter) while breaking up any agglomerates of mutually adherent residue granules that may have formed in the mass, i.e. to enable expulsion thereof through the aperture 16. The lower end of the mass 12 may be cooled, in accordance with procedures heretofore proposed, as by heat exchange with a relatively cool supplemental flow of trichloride gas introduced to the lower end of the converter through suitable means (not shown), e.g. to cool the residue to an exit temperature between about 300° C. and about 600° C. It will be understood, of course, that the above-described details of converter structure, cone, discharge aperture configuration and the like are shown and set forth for illustrative purposes only, and that the present invention may be employed with alternative forms of such structures and devices, e.g. in association with converter discharge apertures of annular or other configuration.

As employed with the converter 10, the discharge apparatus shown in FIG. 1 includes an upright elongated hollow shaft or pipe 18, having an upper cylindrical section 19 and a lower cylindrical section 20, larger in diameter than the section 19, joined by a downwardly flaring frusto-conical section 21. The upper end of the pipe 18 communicates with the converter discharge aperture 16, so as to receive residue alloy expelled through the latter aperture. The lower end of the pipe 18 opens into a housing 22; a rotary table feeder 23 is positioned immediately beneath the open pipe end in the housing 22 to receive residue alloy advancing downwardly through the pipe 18 from the aperture 16.

This feeder 23, which may be entirely conventional in structure and operation, is rotated by suitable means (not shown) to carry the residue received thereon past a stationary peripheral plough 23a which is disposed to

scrape the residue from the feeder into the upper end of a further upright pipe 24. The pipe 24 extends downwardly from the housing 22 into a body of water 25 contained in a downwardly tapering closed tank 26; specifically, the pipe 24 opens into the tank 26 at a level sufficiently far below the water level in the tank so that water stands in the lower end of pipe 24 to a level indicated at 30. Level 30 is lower than level 28 because the pressure in pipe 18, housing 22 and pipe 24 corresponds to gas pressure in the converter chamber 11, which as stated is ordinarily above atmospheric pressure, while the gas pressure in tank 26 ordinarily equals atmospheric pressure.

In the illustrated arrangement, the pipe 18, housing 22, and pipe 24 together constitute a continuous, laterally confined (i.e. gas-tight) passage for advance of residue alloy from the converter discharge aperture 16 into the body of water 25; the water level 30 in pipe 24 provides a water seal effective to prevent flow of gas either into or out of the lower end of the passage. The apparatus further includes means, shown schematically as spray heads 32, positioned in the pipe 24 at a substantial distance above the water level 30 for directing sprays of water onto the residue alloy descending in the latter pipe, water being supplied to the spray heads through conduits 34. In addition, a conduit 35 controlled by a valve 36 opens into the lower portion of the pipe section 20 for effecting supply of steam or water to the section 20; and a conduit 37 controlled by a valve 38 communicates with the upper portion of pipe section 20 for supplying purge gas to, or withdrawing purge gas from, the pipe 18.

Residue delivered into the body of water 25 through pipe 24 is discharged from the tank 26 by an upwardly inclined screw conveyor 40, driven by suitable means (not shown) and having a housing 42. The lower end of the screw conveyor opens into the body of water 25 below the lower end of pipe 24, to receive residue alloy accumulating at the bottom of tank 26; the upper end of the screw conveyor opens at a locality above and external to the tank, to discharge the residue to the open air. The tank 26 is also provided with an exhaust duct 45 (opening into the tank above the water level 28 therein) for venting to the atmosphere gases generated in the tank by reaction of the water with the residue alloy.

In performing the process of the present invention with the foregoing apparatus, residue alloy in the form of lumps and granules e.g. at a temperature between about 300° C. and about 600° C. is expelled by the cone 14 from the converter 10 into the upper end of the pipe 18 and falls freely through the upper section 19 into the lower section 20. Initially the residue is permitted to accumulate in the section 20, i.e. on and above the table feeder 23, until the section 20 is filled to a predetermined level adjacent its upper extremity with a loose, gas-permeable body or column 47 of the residue lumps and granules. Thereafter the feeder 23 is rotated, to withdraw residue from the lower end of the section 20 and thus to effect downward advance of the residue in the column 47, at a rate controlled to maintain the top of the column at such predetermined level, i.e. to maintain the section 20 substantially filled with this column of relatively hot residue expelled from the converter.

From the pipe section 20, the residue is delivered by table feeder 23 to the pipe 24 and falls freely there-through into the water in the tank 26. As the residue descends through the portion of the latter pipe extending between the spray heads 32 and the water level 30, it comes into contact with the cooling sprays of water from the spray heads 32 and is thus cooled before it enters the body of water at the lower end of the pipe 24. The residue accumulates in the tank 26 below the pipe 24 and is discharged from the tank to the open air by the screw conveyor 40, e.g. for removal to a disposal area

as by means of a conveyor belt 48. Water is withdrawn from the tank 26 through conduit 49, and may be cooled and returned to the system through conduits 34.

In the pipe 24, gas (principally methane, as explained above, together with some hydrogen) is generated upon contact of the residue with the cooling sprays, by reaction of the water-active constituents of the residue. Further generation of such gas occurs by reaction of water and residue constituents in the body of water 25; as will be appreciated, some of the gas produced in the body of water passes into the pipe 24 and the remainder passes into the upper region of the tank 26 from which it is discharged to the atmosphere through exhaust duct 45.

The gas thus produced in pipe 24 is saturated with water vapor from the cooling sprays and the body of water 25; the amount of such water vapor present in the gas is dependent on the vapor pressure (and thus on the temperature) of the water in the pipe 24. As the produced gas containing water vapor rises in the residue discharge passage to and through the pipe section 20, the water vapor reacts with the hot residue in the column 47 to produce further quantities of methane and hydrogen. Retention of the heated residue as a column in the section 20 affords a contact time sufficient for substantially complete reaction of the water vapor so that the atmosphere in the pipe 18 above the column 47 is essentially free of water vapor.

Accordingly it will be seen that the residue descending in the discharge passage defined by pipe 18, housing 22 and pipe 24 successively traverses a water vapor reacting zone, a cooling zone, and a water seal zone, and that in each of these zones methane and hydrogen gas is produced by contact of the residue with water or water vapor. More particularly, the pipe section 20 filled with the column 47 constitutes the water vapor reacting zone of the passage; the portion of pipe 24 between the spray heads 32 and water level 30 is the cooling zone; and the portion of the path of residue descent extending below the water level 30 is the water seal zone.

Aluminum trichloride gas from the converter enters the upper pipe section 19 through the discharge aperture 16. In the section 19, the trichloride encounters the methane and hydrogen gases produced in the pipe 24 and the column 47, and rising therefrom through and above the column; since these gases above the column 47 are essentially water-free, as stated, no significant formation of hydrochloric acid occurs upon contact of the gases with the trichloride in the section 19. However, if the trichloride gas reached the water vapor in and below the column 47, acid formation would occur, creating a corrosive condition; and if the trichloride gas descended to the body of water 25, it would not only react therewith to produce further acid but would also dissolve in the water, resulting in loss of trichloride from the subhalide distillation system.

To prevent access of the aluminum trichloride gas to the water vapor in and below the pipe section 20 or to the body of water 25, a purge atmosphere effective to prevent downflow of trichloride gas beyond the section 19 is established and maintained in the residue discharge passage. This atmosphere is provided at least in part by the generation of methane and hydrogen in the pipe 24 and column 47. Depending upon operating conditions, as hereinafter further explained, the production of methane and hydrogen gas in the cooling and water seal zones in pipe 24 as described above (together with the production of such gases in the column 47 by reaction of water vapor from the pipe 24) may provide the full amount of purge gas required for this purpose, or indeed in some circumstances may provide an excess of purge gas; in the latter case, the excess purge gas is withdrawn from the pipe 18 through conduit 37. In other circumstances, the amount of gases thus produced may be insufficient to block downflow of trichloride gas beyond the section 19 and in this event additional purge gas as needed to pro-

vide the requisite purge atmosphere may be supplied to the pipe 18 through the conduit 37. The supplied gas may be hydrogen and/or methane (which are ordinarily available from other parts of the subhalide distillation system), or other gas or gases inert with respect to the aluminum trichloride gas and the aluminum in the alloy in the converter.

The rate of production of gas in the cooling and water seal zones is dependent in particular on the content of aluminum carbide and free aluminum in the residue, and the temperature to which the residue is cooled in the cooling zone. Under ordinary operating conditions, if the latter temperature is above 60° C., the amount of water vapor rising through the discharge passage is excessive and production of methane in the passage is much larger than required; accordingly it is presently preferred to cool the residue in the pipe 24 (i.e. by means of the cooling sprays) at least to about 60° C. With cooling of the residue to between about 50° C. and about 60° C., if the residue is rich in aluminum carbide (as in the case of residue expelled from the converter during start-up of the subhalide distillation system) more than enough purge gas to block downflow of aluminum trichloride from the converter may be produced in the pipe 24; on the other hand, if the residue is highly extracted, i.e. very low in aluminum content, production of purge gas (with cooling of the residue to the latter temperature range) may be insufficient to block such downflow. When the residue is cooled to a temperature below about 50° C., gas production in the pipe 24 becomes negligible in the case of most residues.

Thus the purge atmosphere required in the passage to prevent downflow of aluminum trichloride gas beyond the pipe section 19 can be provided by generation of gas in the pipe 24 as described above, with addition or removal of purge gas through conduit 37 as necessary to compensate for particular conditions (such as proportionate aluminum content in the residue) providing low or high rates of gas production in the pipe 24.

Alternatively, and preferably for ease of control of purge gas production, the cooling operation in the pipe 24 may be performed under such conditions that very little vaporization or generation of gas occurs therein, i.e. by providing water sprays effective to cool the residue rapidly to a temperature for example somewhat below 50° C. The purge atmosphere is then provided by supplying steam to the lower portion of the water vapor reacting zone in the pipe section 20 through conduit 35. This steam, rising through the column 47, reacts with the hot residue in the column to produce methane and hydrogen. Specifically, the steam is supplied at such rate as to provide sufficient generation of these gases in the column to establish and maintain in the discharge passage a purge atmosphere effective to prevent downflow of trichloride gas beyond the pipe section 19; the rate of steam supply is also controlled to ensure essentially complete reaction of the steam in the column (so that the gas above the column is essentially moisture-free) and to prevent excessive generation of purge gas in the column. The steam supply rate can readily be regulated by adjustment of the valve 36, e.g. in accordance with variation in the aluminum content of the residue or other factors affecting generation of purge gas in the column 47, thereby enabling close control of the production of purge gas.

As another way of supplying steam to the water vapor reacting zone, water (i.e. in liquid state) may be introduced through conduit 35, in place of steam as described above. Upon contact with the hot residue alloy in the pipe section 20, this water evaporates, generating steam which rises through the column 47 and reacts with the residue to produce methane and hydrogen. The desired control of production of purge gas in this instance is effected by regulating the rate of supply of water through conduit 35.

It will be understood that the dimensions of the pipe

section 20 and the vertical extent of the column 47 therein (determined by the rate of rotation of the feeder 23) are selected to provide a sufficient body of residue to ensure the desired essentially complete reaction of water vapor rising from the pipe 24, and also, in the case of purge gas generation by supply of steam as just described, to enable production of the requisite purge gas.

The purge atmosphere in the residue discharge passage, established in any of the foregoing ways, extends through the passage from the water level 30 upwardly through the column 47 and into the pipe section 19 where, as stated, it encounters and balances the aluminum trichloride atmosphere of the converter to block downflow of trichloride beyond the latter section; as will be apparent, the pressure of this purge atmosphere throughout the passage is substantially equal to the trichloride gas pressure in the converter. Preferably the gas-balancing section of the passage (i.e. the pipe portion between the aperture 16 and the water vapor reacting zone in column 47) is of substantial vertical extent to allow for variations in the level at which the gases balance due to pressure variations in the system.

It will further be apparent that there is some flow of purge gas from the discharge passage into the converter through aperture 16. It is desirable to minimize such flow of purge gas into the converter, by providing a purge supply not substantially more than sufficient to balance the trichloride gas in the pipe section 19, since the purge gas dilutes the trichloride gas flow in the converter; however, as methane and hydrogen are chemically inert with respect to aluminum trichloride gas and aluminum, no adverse reactions are produced by presence of such gases in the converter. The methane reacts in the converter to yield hydrogen which passes (together with the hydrogen constituent of the purge gas) from the converter in the outlet gas flow, and this hydrogen may be removed from the circulating trichloride flow of the subhalide distillation system at any convenient point therein, e.g. downstream of the aforementioned decomposer region, to prevent buildup of hydrogen content in the circulating flow.

Another arrangement of apparatus with which the present process may be practiced is illustrated diagrammatically in FIG. 2. This apparatus, like that of FIG. 1, is shown as associated with a subhalide converter 10 of the type already described, and includes a vertical hollow shaft or pipe 18 communicating at its upper end with the converter residue discharge aperture 16 to receive residue lumps and granules expelled from the lower end of the alloy mass 12 in the converter chamber 11 by the extractor cone 14. As in FIG. 1, the pipe 18 has an upper cylindrical section 19 and a lower cylindrical section 20 (larger in diameter than the section 19) joined by a downwardly flaring frustoconical section 21.

The apparatus of FIG. 2 further includes a downwardly tapering closed tank 50 containing a body of water 51. Integral with the tank structure, and extending downwardly from the central portion of the top of the tank into the body of water 51, is an axially vertical pipe 52 which is open at both ends and has a lower cylindrical portion 53 of internal diameter e.g. about equal to the diameter of the pipe section 20. The upper portion 54 of the pipe 52 flares outwardly to provide a region above the cylindrical portion 53 of upwardly increasing diameter larger than the diameter of the portion 53.

The pipe 18 opens downwardly into the pipe 52 and is in coaxial relation thereto, so that residue alloy descending in the pipe 18 advances into the cylindrical pipe portion 53. As shown, the lower end of the pipe 18 extends into the region defined by the portion 54 of pipe 52 but is spaced away from the latter pipe portion to provide an annular opening 55 between the pipe 18 and the pipe portion 54. Surrounding the lower extremity of the pipe 18 is an annular perforated conduit 57 which is adapted to direct a spray of water (supplied to the conduit 57 through inlet conduit 58) into the opening 55 and

thus into the path of residue descending from the pipe 18 to the pipe portion 53. The opening 55 and annular conduit 57 are enclosed by a suitable housing 60 extending between the pipe 18 and the top of the tank 50 to prevent escape of gases from the pipe 18 and pipe portion 53 through the opening 55.

Accordingly it will be seen that the pipe 18 and pipe 52 in cooperation with the housing 60 define a continuous, laterally confined (i.e. gas-tight) passage for advance of a residue alloy from the converted discharge aperture 16 into the body of water 51. The pipe 52 opens downwardly into the water at a level sufficiently far below the level of water 62 in the tank 50 so that water stands in the lower portion 53 of pipe 52 to a level 64 therein providing a water seal effective to prevent flow of gas either into or out of the lower end of the pipe 52. As in the apparatus of FIG. 1, the level 64 is ordinarily lower than the level 62 because the gas pressure in the pipe 52 corresponds to that in the converter, which is ordinarily higher than ambient atmospheric pressure, whereas the pressure in tank 50 is substantially equal to atmosphere pressure.

The apparatus of FIG. 2 also includes a bucket elevator 66, e.g. of conventional design and powered by suitable means (not shown), which extends from a locality in the tank 50 below the pipe 52, through an opening 67 in the top of the tank, and is adapted to transport the residue from the body of water 51 to a locality external to the tank for discharge into the open air. Again as in the apparatus of FIG. 1, a conduit 35 opening into the lower portion of the shaft section 20 and controlled by a valve 36 is provided for supplying steam or water to the section 20; and a conduit 37 controlled by a valve 38 and opening into the pipe 18 adjacent the upper portion of the section 20 is provided for supplying purge gas to, or removing purge gas from the pipe 18. Gases evolved in the tank 50 are vented to the atmosphere through an exhaust duct 68.

In the process of the invention as performed with the apparatus of FIG. 2, residue alloy expelled from the converter 10 is initially allowed to accumulate in the discharge passage defined by pipe 18 and pipe 52, in and above the water in tank 50, until the passage is filled with a loose, gas-permeable body or column 69 of the residue to a predetermined level substantially above the water level 64, i.e. to a level adjacent the upper extremity of shaft section 20. The bucket elevator 66 is then operated to withdraw the residue from the lower end of column 69 in the tank 50 at a rate controlled to maintain the discharge passage filled with the column 69 of residue lumps and granules extending from the tank 50 upwardly through the pipe 52 and pipe section 20 to the aforementioned level. In this way, the residue expelled from the converter is advanced downwardly through the discharge passage into the body of water 51 and is transferred therefrom to the open air by the bucket elevator, e.g. for removal to a disposal area by a conveyor belt 70. Water is withdrawn from the tank 50 through conduit 71, for cooling and recirculation to the system through conduit 58.

As in FIG. 1, the residue thus descending in the discharge passage successively traverses a water vapor reacting zone, a cooling zone, and a water seal zone. Specifically, the water vapor reacting zone of the passage is the pipe section 20 containing the upper portion of column 69, in which the residue is at a relatively high temperature. The cooling zone is the portion of pipe 52 extending between the annular opening 55 and the water level 64; as the residue traverses this zone in the column 69 it is cooled by contact with sprays of water from the annular conduit 57, i.e. before it enters the body of water. The portion of the path of residue descent extending below the water level 64 constitutes the water seal zone. Methane, together with some hydrogen, is produced in the cooling and water seal zones by reaction of the cooling sprays and the body of water 51 with water-active con-

stituents of the residue; this gas is saturated with water vapor (from the body of water and the cooling sprays), which rises with the gas through the water vapor reacting zone and is essentially completely reacted with the residue therein so that the gas in the pipe section 19 above the latter zone is essentially free of water vapor.

To prevent downflow of aluminum trichloride gas from the converter beyond the pipe section 19, a purge atmosphere effective to block such downflow is established and maintained in the discharge passage, extending from the water level 64 through the column 69 and into the pipe section 19 where it encounters the halide gas from the converter. As in the apparatus of FIG. 1, this purge atmosphere may be provided by the methane and hydrogen gases produced in the cooling and water seal zones, with addition or removal of purge gas through the conduit 37 as necessary to compensate for inadequate or excessive generation of such gas; or alternatively the residue may be rapidly cooled by the cooling sprays to a comparatively low temperature (e.g. below 50° C.) at which very little vaporization or generation of gas occurs, and the major portion of the purge atmosphere may in such case be provided by supplying steam (or water) to the lower portion of pipe section 20 through conduit 35 for reaction with the hot residue in the column 69 to produce methane and hydrogen. As will therefore now be understood, the performance of the present process in the apparatus of FIG. 2 is essentially as described above in connection with FIG. 1, except that in the system of FIG. 2 the residue column extends downwardly beyond the water vapor reacting zone into and through the cooling and water seal zones of the discharge passage; the water vapor reacting zone is, accordingly, contiguous to the cooling zone; and the level of the column in the shaft section 20 is controlled by the rate of discharge of residue from the tank 50.

The following example, employing apparatus of the type shown in FIG. 2, will serve further to illustrate the performance of the process of the present invention:

Residue alloy, in the form of granules and larger lumps ranging e.g. up to 3 inches in size, is expelled at a rate of 3 tons per hour into the upper end of the discharge passage of the apparatus from the converter of a subhalide distillation system. The latter system produces 3 tons per hour of aluminum metal; 30 tons per hour of aluminum trichloride gas are supplied to the converter, for operation at a conversion of 0.25 (i.e. 25% of the supplied gas being converted to aluminum monochloride by reaction with aluminum in the converter alloy charge).

The residue, descending in the discharge passage, is advanced through a water vapor reacting zone and a cooling zone into a water seal zone provided by a body of water in a closed tank at the lower end of the passage, and is discharged from the tank to the open air at a rate of 3 tons per hour, in such manner as to maintain in the passage a column of the residue extending upwardly from the lower end of the passage through the water vapor reacting zone. In and below the latter zone, the discharge passage has a diameter of 18 inches; the total combined vertical extent of the cooling zone and water seal zone in this passage is about 20 feet. The operating pressure in the passage is between 1.2 and 1.5 atm., and hence the water level in the tank (i.e. outside the passage) may be up to 15 feet higher than that in the passage.

The descending residue is cooled by sprays of water in the cooling zone; the temperature of these sprays and of the water in the tank is maintained at 60° C. Methane, together with some hydrogen, is produced in the cooling and water seal zones of the passage at a rate dependent on the aluminum content of the residue. With residue from which up to about 90% of the available aluminum has been extracted in the converter, the rate of methane production in the discharge passage is approximately 400 s.c.f.h. (standard cubic feet per hour); in the case of residue from which more than 90% of the aluminum has been extracted, the rate of methane production is lower,

e.g. decreasing to less than 20% of the aforementioned value. Gas is produced in the tank (outside the discharge passage) at approximately the same rate as in the passage and is vented from the tank to the atmosphere.

The gas thus produced in the cooling and water seal zones is saturated with water vapor at the temperature of operation therein (i.e. 60° C.). At the latter temperature the partial pressure of water is about 150 mm. of mercury. As the gas rises in the passage, the water vapor is removed by reaction with the residue in the column in the water vapor reacting zone such residue being at a temperature of about 200° C.; a five-foot depth (vertical extent) of the residue column in the water vapor reacting zone is effective to remove substantially all the moisture in the gas.

An upward flow of approximately 150 s.c.f.h. of purge gas in the discharge passage is required to prevent downflow of aluminum trichloride gas from the converter into the water vapor reacting zone. The gas-balancing portion of the discharge passage (i.e. the passage portion extending between the converter outlet and the latter zone) is about 10 feet in height, to allow for the effect of pressure variations in the system. Purge gas passing upwardly through the balancing portion enters the converter where it reacts to form hydrogen, providing in the converter atmosphere a hydrogen content of about 0.2 mol percent which is subsequently removed from the recirculating trichloride flow of the subhalide distillation system.

With residue from which up to about 90% of the available aluminum has been removed, the rate of methane production (as set forth above) is about 250 s.c.f.h. greater than necessary to prevent downflow of aluminum trichloride gas; in such case, the excess purge gas is withdrawn from the lower end of the gas-balancing portion of the discharge passage. With residue from which 95% of the aluminum has been removed, the rate of methane production is only about 80 s.c.f.h., which is less than that necessary to balance the trichloride gas; addition of an approximately equal amount (80 s.c.f.h.) of hydrogen as purge gas at the lower end of the gas-balancing portion of the discharge passage satisfactorily prevents access of aluminum trichloride to the water vapor reacting zone.

It is to be understood that the invention is not limited to the procedures and embodiments hereinabove specifically set forth, but may be carried out in other ways without departure from its spirit.

We claim:

1. In a method of removing residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of advancing successive quantities of said residue alloy from said converter region into and through a laterally confined passage and supplying purge gas to said passage to provide a purge atmosphere extending through said passage to encounter aluminum halide gas entering said passage from said converter region, and effective to prevent flow of aluminum halide gas from said converter region beyond a predetermined first locality in said passage, said step of supplying purge gas including exposing said alloy to water at a second locality in said passage, spaced from said first locality in the direction of advance of said alloy, to produce gas providing at least a part of said purge atmosphere by reaction of the water with water-active constituents of the alloy.

2. In a method of removing residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of advancing successive quantities of said residue alloy from said converter region into and through a laterally confined passage so that a gas-permeable column of said alloy is maintained in at least a portion of said passage intermediate first and second localities therein successively traversed by said alloy, and supplying purge gas to said passage to provide a purge atmosphere extending through said passage to encounter

aluminum halide gas entering said passage from said converter region and effective to prevent flow of aluminum halide gas from said converter region beyond said first locality in said passage, said step of supplying purge gas including exposing said alloy to water at said second locality in said passage to produce gas providing at least a part of said purge atmosphere by reaction of the water with water-active constituents of the alloy, said last-mentioned gas advancing in said passage from said second locality through said column toward said converter region together with water vapor, and said column providing a mass of alloy intermediate said first and second localities sufficient to effect essentially complete removal of water vapor from said last-mentioned gas advancing therethrough, by reaction of said water vapor with water-active constituents of the alloy in said mass.

3. In a method of removing residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of discharging successive quantities of said residue alloy from said converter region into a laterally confined passage extending downwardly from said converter region, advancing said quantities of alloy downwardly through said passage so that a gas-permeable column of said alloy is maintained in at least a portion of said passage filling said passage portion at least to a predetermined level therein, and supplying purge gas to said passage to provide a purge atmosphere extending through said passage to encounter aluminum halide gas entering said passage from said converter region and effective to prevent downflow of aluminum halide gas from said converter region to said predetermined level in said passage, said step of supplying purge gas including exposing said alloy to water at a locality in said passage below said predetermined level to produce gas providing at least a part of said purge atmosphere by reaction of the water with water-active constituents of the alloy, said last-mentioned gas rising through said column together with water vapor, and said column providing a mass of alloy intermediate said last-mentioned locality and said predetermined level sufficient to effect essentially complete removal of water vapor from said last-mentioned gas rising therethrough, by reaction of the water vapor with water-active constituents of the alloy in said mass.

4. In a method of removing granular residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of discharging successive quantities of said granular residue alloy from said converter region into a laterally confined passage extending downwardly from said converter region, advancing said quantities of alloy downwardly through said passage so that a gas-permeable column of said alloy is maintained in at least a portion of said passage filling said passage portion at least to a predetermined level therein spaced downwardly from said converter region, and exposing said alloy to water at a locality in said passage below said predetermined level to produce purge gas, by reaction of the water with water-active constituents of the alloy, providing a purge atmosphere extending through said passage above said predetermined level to encounter aluminum halide gas entering said passage from said converter region and effective to prevent downflow of aluminum halide gas from said converter region to said predetermined level in said passage, said purge gas rising through said column together with water vapor, and said column providing a mass of alloy intermediate said last-mentioned locality and said predetermined level sufficient to effect essentially complete removal of water vapor from said last-mentioned gas rising therethrough, by reaction of the water vapor with water-active constituents of the alloy in said mass.

5. A method according to claim 4, wherein said step of exposing said alloy to water comprises introducing

steam to said passage at said locality to provide an upward flow of steam through said column for reaction of said steam with water-active constituents of the alloy in said column to produce said purge gas, said steam being introduced to said passage at a rate selected to provide sufficient production of gas in said column to prevent downflow of aluminum halide gas from said converter region to said predetermined level in said passage.

6. In a method of removing residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of discharging successive quantities of said residue alloy from said converter region into a laterally confined passage extending downwardly therefrom, advancing said quantities of alloy downwardly through said passage so that at least a portion of said passage is maintained filled with a gas-permeable column of said alloy at least to a predetermined level therein, and delivering said quantities of alloy into a body of water maintained at the lower end of said passage and providing a water seal in said passage at a locality below said predetermined level, while maintaining in said passage a purge atmosphere extending through said passage to encounter aluminum halide gas entering said passage from said converter region and effective to prevent downflow of said aluminum halide gas to said predetermined level, at least a part of said atmosphere being provided by gas produced by reaction of water with water-active constituents of said alloy in said passage below said predetermined level, said last-mentioned gas rising in said passage through said column together with water vapor, and said column providing a mass of alloy intermediate said last-mentioned locality and said predetermined level sufficient to effect essentially complete removal of water vapor from the gas rising therethrough, by reaction of the water vapor with water-active constituents of the alloy in said mass.

7. In a method of removing residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of discharging successive quantities of said residue alloy from said converter region into a laterally confined passage extending downwardly therefrom, advancing said quantities of alloy downwardly through said passage so that at least a portion of said passage is maintained filled with a gas-permeable column of said alloy at least to a predetermined level therein, delivering said quantities of alloy into a body of water maintained at the lower end of said passage and providing a water seal in said passage at a first locality below said predetermined level, and directing sprays of water into the path of said alloy in said passage at a second locality intermediate said predetermined level and said first locality for contact with said alloy to cool said alloy, while maintaining in said passage a purge atmosphere extending through said passage to encounter aluminum halide gas entering said passage from said converter region and effective to prevent downflow of said aluminum halide gas to said predetermined level, at least a part of said atmosphere being provided by gas produced by reaction of said sprays of water and said body of water with water-active constituents of said alloy, said last-mentioned gas rising in said passage through said column together with water vapor, and said column providing a mass of alloy intermediate said second locality and said predetermined level sufficient to effect essentially complete removal of water vapor from the gas rising therethrough, by reaction of the water vapor with water-active constituents of the alloy in said mass.

8. In a method of removing granular residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of discharging successive quantities of said granular alloy from said converter region into a laterally confined passage extending downwardly

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therefrom into a body of water maintained at the lower end of said passage and providing in said passage a water seal at a first locality adjacent said lower end thereof, advancing said quantities of alloy downwardly through a portion of said passage intermediate said converter region and said first locality at a rate controlled to maintain said passage portion filled with a gas-permeable column of said alloy at least to a predetermined level therein spaced downwardly from said converter region, delivering said alloy from said passage portion into said body of water, directing sprays of water into the path of said alloy in said passage at a second locality intermediate said first locality and said predetermined level for contact with said alloy to cool said alloy, said sprays of water and said body of water being at a temperature of not more than about 60° C., while maintaining in said passage a purge atmosphere extending through said passage to encounter aluminum halide gas entering said passage from said converter region and effective to prevent downflow of said aluminum halide gas to said predetermined level, at least a part of said atmosphere being provided by gas produced by reaction of water with water-active constituents of said alloy in said passage below said predetermined level, said last-mentioned gas rising in said passage through said column together with water vapor, and said column providing a mass of alloy intermediate said second locality and said predetermined level sufficient to effect essentially complete removal of water vapor from the gas rising therethrough, by reaction of the water vapor with water-active constituents of the alloy in said mass.

9. A method according to claim 8, wherein the temperature of said sprays of water and said body of water is in a range between about 50° C. and about 60° C., and wherein at least a substantial part of said purge atmosphere is provided by gas produced by reaction of said sprays of water and said body of water with water-active constituents of said alloy.

10. A method according to claim 9 including the step of withdrawing purge gas from said passage, to prevent build-up of excess purge gas in said passage, upon production of gas in said passage at a rate in excess of that necessary to prevent downflow of said aluminum halide gas to said predetermined level.

11. A method according to claim 8, wherein said step of maintaining said purge atmosphere in said passage includes the step of introducing to said passage a flow of purge gas inert with respect to aluminum and to said aluminum halide to augment the gas produced in said passage.

12. A method according to claim 8 wherein said sprays of water and said body of water are provided at a temperature for cooling the alloy descending in said passage without substantial production of gas upon contact of said sprays of water and said body of water with the alloy descending in said passage, and wherein said step of maintaining said passage filled with said purge atmosphere comprises supplying steam to said passage at a third locality intermediate said predetermined level and said first locality to provide an upward flow of steam through said column for reaction of said steam with water-active constituents of the alloy in said column to produce gas providing said purge atmosphere, said steam being introduced to said passage at a rate selected to provide sufficient production of gas in said column to prevent downflow of said aluminum halide gas to said predetermined level.

13. In a method of removing granular residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of discharging successive quantities of said granular alloy from said converter region into a laterally confined passage having

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an upper portion extending downwardly from said converter region and a lower portion extending downwardly from said upper portion, transferring said alloy from the lower end of said upper portion to the upper end of said lower portion at a rate controlled to maintain said upper portion filled with a gas-permeable column of said alloy at least to a predetermined level therein spaced downwardly from said converter region delivering said alloy through said lower portion into a body of water maintained at the lower end of said lower portion and providing a water seal in said lower portion at a first locality adjacent said lower end thereof, and directing sprays of water into the path of said alloy in said lower portion at a second locality above said first locality for contact with said alloy to cool said alloy, while maintaining in said passage a purge atmosphere extending through said passage to encounter aluminum halide gas entering said passage from said converter region and effective to prevent downflow of said aluminum halide gas to said predetermined level, at least a part of said atmosphere being provided by gas produced by reaction of said sprays of water and said body of water with water-active constituents of said alloy, said last-mentioned gas rising in said passage through said column together with water vapor, and said column providing a mass of alloy below said predetermined level in said upper passage portion sufficient to effect essentially complete removal of water vapor from the gas rising therethrough, by reaction of the water vapor with water-active constituents of the alloy in said mass.

14. In a method of removing granular residue alloy from an aluminum subhalide distillation system having a converter region wherein said alloy is heated and exposed to aluminum halide gas, the steps of discharging successive quantities of said granular alloy from said converter region into a laterally confined passage extending downwardly therefrom into a body of water maintained at the lower end of said passage and providing in said passage a water seal at a first locality adjacent said lower passage end, for delivery of said alloy into said body of water, withdrawing said alloy from said body of water at the lower end of said passage at a rate controlled to maintain said passage filled with a gas-permeable column of said alloy at least to a predetermined level therein above said first locality, and directing sprays of water into said column at a second locality intermediate said first locality and said predetermined level for cooling said alloy, while maintaining in said passage a purge atmosphere extending through said passage to encounter aluminum halide gas entering said passage from said converter region and effective to prevent downflow of said aluminum halide gas to said predetermined level, at least a part of said atmosphere being provided by gas produced by reaction of said sprays of water and said body of water with water-active constituents of said alloy, said last-mentioned gas rising in said passage through said column together with water vapor, and said column providing a mass of alloy intermediate said second locality and said predetermined level sufficient to effect essentially complete removal of water vapor from the gas rising therethrough, by reaction of the water vapor with water-active constituents of the alloy in said mass.

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