

21346
Singapore

PATENT SPECIFICATION

(11) 1271521

DRAWINGS ATTACHED

- (21) Application No. 43146/68 (22) Filed 11 Sept. 1968
(23) Complete Specification filed 11 Aug. 1969
(45) Complete Specification published 19 April 1972
(51) International Classification B01D 53/26
(52) Index at acceptance B1L 11A
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(54) TREATMENT OF CHLORINE

(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED of Imperial Chemical House, Millbank, London, S.W.1., a British Company do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the treatment of impure moist chlorine.

The treatment of chlorine for liquefaction and use in a number of chemical processes has hitherto required an elaborate and costly system for cleaning, cooling and drying the chlorine gas prior to compression in large complex compressors. To prevent corrosion of the materials of construction of the compressor by the dry chlorine, it is necessary to prevent the temperature of the gas from rising to more than about 120°C. In order to do this it is necessary to restrict the compression ratio at each compression stage to about 2.5 to 1 and to cool the gas between such stages.

In a large chlorine production unit, the cleaning, cooling and drying equipment and pipework is of large volume owing to its being on the low pressure side of the compressor. The treatment of gases at these sub-atmospheric pressures requires the use of low pressure-drop equipment involving circulating liquid systems incorporating pumps and coolers.

We have now found that these disadvantages can be largely overcome by compressing the impure chlorine gas before it is cleaned and dried.

Thus according to the invention there is provided a process for the treatment of impure moist chlorine gas which comprises (a) compressing said gas in a compressor in which at least those parts which come in contact with the chlorine are made of titanium or an alloy of titanium and in which the compressor is cooled internally and/or sealed with an aqueous solution of an alkali metal chloride

or an alkaline earth metal chloride and (b) subsequently drying the compressed moist chlorine.

According to another aspect of the invention there is provided an apparatus for the treatment of impure moist chlorine gas comprising in combination a compressor followed by a bubble plate scrubber, the compressor having at least those parts which come into contact with the chlorine made from titanium or an alloy of titanium and being sealed with an aqueous solution of an alkali or alkaline earth metal chloride.

The process finds application in the treatment of impure moist chlorine obtained by a wide variety of methods, but it is particularly applicable in the treatment of chlorine obtained by electrolysis of solutions of alkali metal chlorides, in particular sodium chloride brine.

The compressor is sealed and/or cooled with an aqueous solution of an alkali metal chloride, e.g. sodium chloride or alkaline earth metal chloride, e.g. calcium chloride. The use of solutions of these chlorides reduces the tendency of chlorine hydrate to be formed compared with sealing and/or cooling with water.

In a preferred embodiment of our process the chlorine gas is cooled, after compression by contact with aqueous solution of an alkali metal chloride (for example, a sodium chloride brine) or an aqueous solution of an alkaline earth metal chloride (for example, calcium chloride solution). Alkali metal chloride brine used for irrigating or cooling may be fed to the electrolytic cells in which the chlorine gas is produced. In one example of the invention, brine or water spray may be removed from the gas before drying by means of a filter made from a synthetic fibre, especially a polyester fibre, for example that sold under the name "Terylene" ("Terylene" is a Registered Trade Mark).

The compressed chlorine gas is be dried, for example, with concentrated sulphuric acid.

[Price 25p]

Since drying of the compressed chlorine may be carried out at a higher pressure than is possible when drying is done prior to compression, more compact equipment can be used, for example, a bubble-plate scrubber. Gas drying may be accomplished by sulphuric acid in a single tray type column, which may be constructed mainly of mild steel. The sulphuric acid used for drying may be recirculated for cooling or used directly without external cooling. The acid mist may be removed from the chlorine gas after drying by means of a glass fibre filter. Examples of the materials of construction of the sulphuric acid trays are polyvinyl chloride; chlorinated polyvinyl chloride; a phenolic resin reinforced with asbestos; a ceramic material or glass.

It is preferred to compress the gas to a pressure of between 50 pounds per square inch absolute and 250 pounds per square inch absolute. A particularly convenient pressure range is from 100 pounds per square inch absolute to 150 pounds per square inch absolute.

The invention is illustrated but not limited by the following example with reference to the drawing accompanying the provisional specification, which shows diagrammatically one form of apparatus in which the invention may be put into practice.

Pipe 1 leads directly from the chlorine off-take system of an electrolytic cell to a titanium rotary screw compressor 2 which is driven by an electric motor 3 through a gearbox 4. The gas off-take 5 from the compressor 2 leads to the base of a composite column 6. This column consists, in ascending order, of a lower section 7 for collecting brine used in irrigating the compressor; a ring-packed cooling section 8 down which cold brine flows; a polyester fibre filter 9 to remove brine spray; a bubble-plate sulphuric acid drying section 10 and a glass fibre acid mist filter 11. Pipe 12 is provided for feeding the cooling section 8 with cold brine, pipe 13 for feeding the brine which has passed through the cooling section 8 to the suction side of the compressor via pipe 1, and pipe 14 for conveying the brine collected in section 7 to a flash vessel 15. Pipe 16 is connected either to drain or to the brine feed of the electrolytic cell producing the chlorine (not shown). Pipe 17 returns chlorine gas released in the flash vessel 15 to the compressor via pipe 1. Flash vessel 18 is provided to release chlorine gas from the concentrated sulphuric acid used in drying the compressed gas. The released chlorine is returned to pipe 1 via pipe 19 and 17. An off-take pipe 20 for dry, cooled, compressed chlorine is fitted to the top of the column.

In operation, gas at a temperature of 70°C consisting substantially of chlorine and containing water vapour is drawn at a pressure slightly lower than normal atmospheric pressure directly from an electrolytic cell and passed via pipe 1 to the compressor 2. The

compressor is irrigated by brine fed from the base of the cooling section 8. The impure chlorine gas, now compressed to a pressure of 115 p.s.i.a. is fed to section 7 of column 6 together with the discharged irrigating brine. The compressed gas is now cooled in section 8 and any brine mist entrained in it removed by filter 9. It is now dried by allowing it to make contact with concentrated sulphuric acid in the bubble plate section 10 and removal of acid mist is effected in filter 11. Dry cell gas at a temperature of 30°C and a pressure of 115 p.s.i.a. is drawn off through pipe 20.

WHAT WE CLAIM IS:—

1. A process for the treatment of impure moist chlorine gas which comprises (a) compressing said gas in a compressor in which at least those parts which come in contact with the chlorine are made of titanium or an alloy of titanium and in which the compressor is cooled internally and/or sealed with an aqueous solution of an alkali metal chloride or an alkaline earth metal chloride and (b) subsequently drying the compressed moist chlorine.

2. Process according to Claim 1 wherein the chlorine gas is obtained by the electrolysis of a solution of an alkali metal chloride.

3. Process according to Claim 2 wherein the alkali metal chloride is sodium chloride.

4. Process according to Claim 1, 2 or 3 wherein the cooling and/or sealing liquid is an aqueous solution of sodium chloride.

5. Process according to any one of the preceding claims wherein the compressor is a rotary screw compressor.

6. Process according to any one of the preceding claims wherein the compressed chlorine is cooled with an aqueous solution of an alkali metal chloride or an alkaline earth metal chloride.

7. Process according to claim 6 wherein the alkali metal chloride is sodium chloride.

8. Process according to any one of the preceding claims wherein the alkali metal chloride solution is fed after use for cooling and/or sealing the compressor to electrolytic cells in which the chlorine gas is produced.

9. Process according to any one of the preceding claims wherein water or brine spray is removed from the chlorine gas before drying with a filter made from a synthetic fibre.

10. Process according to Claim 9 wherein the synthetic fibre is a polyester fibre.

11. Process according to any one of the preceding claims wherein the compressed chlorine is dried with sulphuric acid.

12. Process according to Claim 11 wherein the compressed chlorine is dried in a bubble-plate scrubber.

13. Process according to Claim 12 wherein the bubble-plate scrubber has trays made from a plastics material.

14. Process according to Claim 13 wherein the plastics material is polyvinyl chloride,

Since drying of the compressed chlorine may be carried out at a higher pressure than is possible when drying is done prior to compression, more compact equipment can be used, for example, a bubble-plate scrubber. Gas drying may be accomplished by sulphuric acid in a single tray type column, which may be constructed mainly of mild steel. The sulphuric acid used for drying may be recirculated for cooling or used directly without external cooling. The acid mist may be removed from the chlorine gas after drying by means of a glass fibre filter. Examples of the materials of construction of the sulphuric acid trays are polyvinyl chloride; chlorinated polyvinyl chloride; a phenolic resin reinforced with asbestos; a ceramic material or glass.

It is preferred to compress the gas to a pressure of between 50 pounds per square inch absolute and 250 pounds per square inch absolute. A particularly convenient pressure range is from 100 pounds per square inch absolute to 150 pounds per square inch absolute.

The invention is illustrated but not limited by the following example with reference to the drawing accompanying the provisional specification, which shows diagrammatically one form of apparatus in which the invention may be put into practice.

Pipe 1 leads directly from the chlorine off-take system of an electrolytic cell to a titanium rotary screw compressor 2 which is driven by an electric motor 3 through a gearbox 4. The gas off-take 5 from the compressor 2 leads to the base of a composite column 6. This column consists, in ascending order, of a lower section 7 for collecting brine used in irrigating the compressor; a ring-packed cooling section 8 down which cold brine flows; a polyester fibre filter 9 to remove brine spray; a bubble-plate sulphuric acid drying section 10 and a glass fibre acid mist filter 11. Pipe 12 is provided for feeding the cooling section 8 with cold brine, pipe 13 for feeding the brine which has passed through the cooling section 8 to the suction side of the compressor via pipe 1, and pipe 14 for conveying the brine collected in section 7 to a flash vessel 15. Pipe 16 is connected either to drain or to the brine feed of the electrolytic cell producing the chlorine (not shown). Pipe 17 returns chlorine gas released in the flash vessel 15 to the compressor via pipe 1. Flash vessel 18 is provided to release chlorine gas from the concentrated sulphuric acid used in drying the compressed gas. The released chlorine is returned to pipe 1 via pipe 19 and 17. An off-take pipe 20 for dry, cooled, compressed chlorine is fitted to the top of the column.

In operation, gas at a temperature of 70°C consisting substantially of chlorine and containing water vapour is drawn at a pressure slightly lower than normal atmospheric pressure directly from an electrolytic cell and passed via pipe 1 to the compressor 2. The

compressor is irrigated by brine fed from the base of the cooling section 8. The impure chlorine gas, now compressed to a pressure of 115 p.s.i.a. is fed to section 7 of column 6 together with the discharged irrigating brine. The compressed gas is now cooled in section 8 and any brine mist entrained in it removed by filter 9. It is now dried by allowing it to make contact with concentrated sulphuric acid in the bubble plate section 10 and removal of acid mist is effected in filter 11. Dry cell gas at a temperature of 30°C and a pressure of 115 p.s.i.a. is drawn off through pipe 20.

WHAT WE CLAIM IS:—

1. A process for the treatment of impure moist chlorine gas which comprises (a) compressing said gas in a compressor in which at least those parts which come in contact with the chlorine are made of titanium or an alloy of titanium and in which the compressor is cooled internally and/or sealed with an aqueous solution of an alkali metal chloride or an alkaline earth metal chloride and (b) subsequently drying the compressed moist chlorine.
2. Process according to Claim 1 wherein the chlorine gas is obtained by the electrolysis of a solution of an alkali metal chloride.
3. Process according to Claim 2 wherein the alkali metal chloride is sodium chloride.
4. Process according to Claim 1, 2 or 3 wherein the cooling and/or sealing liquid is an aqueous solution of sodium chloride.
5. Process according to any one of the preceding claims wherein the compressor is a rotary screw compressor.
6. Process according to any one of the preceding claims wherein the compressed chlorine is cooled with an aqueous solution of an alkali metal chloride or an alkaline earth metal chloride.
7. Process according to claim 6 wherein the alkali metal chloride is sodium chloride.
8. Process according to any one of the preceding claims wherein the alkali metal chloride solution is fed after use for cooling and/or sealing the compressor to electrolytic cells in which the chlorine gas is produced.
9. Process according to any one of the preceding claims wherein water or brine spray is removed from the chlorine gas before drying with a filter made from a synthetic fibre.
10. Process according to Claim 9 wherein the synthetic fibre is a polyester fibre.
11. Process according to any one of the preceding claims wherein the compressed chlorine is dried with sulphuric acid.
12. Process according to Claim 11 wherein the compressed chlorine is dried in a bubble-plate scrubber.
13. Process according to Claim 12 wherein the bubble-plate scrubber has trays made from a plastics material.
14. Process according to Claim 13 wherein the plastics material is polyvinyl chloride,



- chlorinated polyvinyl chloride or a phenolic resin reinforced with asbestos.
15. Process according to Claim 13 wherein the bubble-plate scrubber is made from a ceramic material or glass.
16. Process according to any one of the preceding Claims wherein the chlorine gas is compressed to a pressure from 50 pounds per square inch absolute to 250 pounds per square inch absolute.
17. Process according to Claim 16 wherein the chlorine gas is compressed to a pressure from 100 pounds per square inch absolute to 150 pounds per square inch absolute.
18. Process for the treatment of impure moist chlorine substantially as hereinbefore described with reference to the Drawing accompanying the provisional specification.
19. Apparatus for the treatment of impure moist chlorine comprising in combination a compressor and followed by a bubble-plate scrubber, the compressor having at least those parts which come into contact with the chlorine made from titanium or an alloy of titanium and being sealed with an aqueous solution of an alkali or alkaline earth metal chloride.
20. Apparatus according to Claim 19 wherein the bubble-plate scrubber has trays made from a plastics material.
21. Apparatus according to Claim 20 wherein the plastics material is polyvinyl chloride, chlorinated polyvinyl chloride or a phenolic resin reinforced with asbestos.
22. Apparatus according to Claim 19 wherein the bubble-plate scrubber has trays made from a ceramic material or glass.
23. Apparatus according to any one of claims 19 to 22 comprising additionally a filter made from a synthetic fibre.
24. Apparatus according to claim 23 wherein the synthetic fibre is a polyester fibre.
25. Apparatus for the treatment of impure moist chlorine constructed and arranged substantially as hereinbefore described and as shown in the Drawing accompanying the provisional specification.
- P. B. TUNNICLIFFE
Agent for the Applicants

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1972.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.



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