[Second Edition.]

PATENT SPECIFICATION.

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433

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COMPLETE SPECIFICATION

Improvements in and relating to the Purification of Gases containing Hydrogen Sulphide.

We, W. C. HOLMES & COMPANY LIMITED, a British Company, of Whitestone Iron Works, Huddersfield, in the County of York, and CHARLES COOPER and DANIEL MAYON HENSHAW, both of the said Company's address and both British Subjects, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The present invention relates to purification of gases containing hydrogen sulphide.

According to the present invention we provide a process for the treatment of coal gas and other gases containing hydrogen sulphide and carbon dioxide, which consists in washing with a dilute solution of ammonium hydroxide in a washer under such conditions that the time of contact of the gases with the liquid is so short that hydrogen sulphide is selectively absorbed without the addition of ammonia from outside the system, characterised by the features that the ammonia from the liquor in the hydraulic main is used to supplement the ammonia in the absorption system and that only about 70—90% of the hydrogen sulphide is absorbed by keeping the ratio of H_2S to NH_3 in the wash liquor between about 1:1 and 1:1.5 parts by weight, while the balance of the hydrogen sulphide is removed by an iron oxide purifier so that the process is substantially self-sustaining.

The ratio of NH_3 to H_2S as recovered in the purification of coal gas is usually about 1 to 2 by weight which corresponds to the bisulphide. The wash liquor leaving the H_2S washer is a solution of normal sulphide plus a little hydrate—and thus contains considerably more NH_3 than is recoverable from the gas. It is possible by distillation to separate the whole of the H_2S from the NH_3 in this liquor, and the H_2S may then be recovered in several ways, e.g.

(a) Combustion with limited air to form sulphur in a Claus kiln.

(b) Combustion with excess air to SO_2. This is absorbed in the original NH_3 solution to form ammonium sulphite, which solution is in turn used to absorb SO_2, giving ammonium bisulphite. Equivalent proportions of NH_3 and H_2S (as SO_2) are thus secured, and the whole of the NH_3 and H_2S are recovered as a single product. This solution is treated with H_2SO_4 to yield sulphate of ammonia and free SO_2, the latter being fed to a chamber or contact acid plant.

The above two methods are well known and are being practised.

(c) To avoid the combustion of the H_2S, the weak ammonium sulphide solution can be distilled so that the whole of the H_2S but only half the NH_3 is evolved. If these gases are condensed with water in a suitable apparatus to form a weak solution (4—5% NH_3), it is possible to produce a solution of NH_4HS and thus recover the NH_3 and H_2S as one product. At higher concentrations some $(NH_4)_2S$ is always present and at 15—20% NH_3 the ratio of $H_2S/NH_3 = 1.5$ by weight. Whichever of these alternatives is adopted may depend upon the location of the sulphate of ammonia and acid plant in which the solution is to be treated. Where this is adjacent to the purifying plant the weak solution of bisulphide may be made. It is preferable however to produce a concentrated product for transport, and then some 25% of the H_2S remains unabsorbed.

This unabsorbed H_2S could be treated separately by iron oxide and being pure H_2S is thereby very easily absorbed; or it may be returned to the gas after NH_3 removal this gas being finally purified by iron oxide purifiers.

Two preferred methods of carrying out the invention are given below and illustrated in the accompanying drawings.

EXAMPLE 1.

The gas after cooling and tar removal is passed through a suitable washer (1) (see Figure 1) in which the time of contact is 1—2 seconds. The usual type of packed scrubber or rotary washer will generally be found unsuitable as the time of contact may be excessive. A bubble cap type of washer is suitable but this must be designed so that there is minimum surface contact between the gas and liquid

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Price 4s 6d

surface in the trays.

The washer is fed with a solution containing 1.5% NH_3 as ammonium hydrate and 92 gallons of wash liquor are required per 13,000 cu. ft. of gas, this being taken as equivalent to 1 ton of coal. The gas entering the washer contains 150 grains NH_3 and 600 grains H_2S per 100 cu. ft. and leaves with the same NH_3 content but only 50 grains of H_2S. The wash liquor leaves with 1.5% NH_3, 1.2% H_2S, and 0.1% CO_2. The gas then passes through a rotary washer (2) where the remaining NH_2 is removed by water, as in common practice.

The liquor leaving the bubble cap washer is treated in a dissociating still (3) whereby substantially all the H_2S and CO_2 are evolved and sufficient NH_3 to give a weight ratio $H_2S/NH_3=2.0$ in the vapours. In order to maintain a constant volume of liquor in circulation the dissociator is so designed that the heating is effected by heat interchange and by closed steam, so that no dilution takes place. The still head temperature is maintained at about 90° C. to give 15—20% NH_3 in the product. The vapours are condensed in a condenser (4) whereby the whole of the NH_3 and part of the H_2S is absorbed. To obtain the maximum possible absorption of H_2S, the cooled condensate passes through a scrubber (5) and the uncondensed gas passes in counter current through this scrubber. This allows further absorption of the H_2S until the H_2S/NH_3 ratio$=1.5$, beyond which no further H_2S can be absorbed. The proportion of undissolved H_2S will depend on the NH_3/H_2S ratio in the crude gas. An average NH_3 content may be taken as 6 lb. per ton of coal, equal to, say, 320 grains per 100 cu. ft. This will absorb 480 grains per 100 cu. ft. of H_2S leaving 120 grains unabsorbed. The concentrated solution of ammonium sulphide and bisulphide may be distilled in the usual way for the manufacture of sulphate of ammonia, the H_2S passing from the saturator to the kilns of the chamber acid plant for conversion into H_2SO_4 or to a separate contact plant.

The unabsorbed H_2S may be treated in several ways.

a) It may be absorbed by iron oxide.

b) It may be burnt to SO_2 in a chamber or contact plant. Alternatively more H_2S may be allowed to pass through the H_2S washer increasing this quantity to 120 grains per 100 cu. ft. The H_2S removed is then 480 grains per 100 cu. ft. and the NH_3 available for combination is 320 grains per 100 cu. ft. giving a ratio of 1.5. The whole of the H_2S in the wash liquor is thus combined with the NH_3 as concentrated liquor.

The liquor leaving the dissociating still (3) is a solution of ammonium hydrate containing 0.82% NH_3. This solution must be strengthened to 1.5% NH_3, which is effected by concentrating the ammonia liquor produced in the removal of NH_3 from the gas in the usual gas works practice. The gas liquor is collected in tank (9) and fed to a dissociator (6) where the H_2S and CO_2 are evolved, these being returned to the crude gas. The liquor then flows via a lime vessel to a free still (7) where the NH_3 is evolved. This is fed to the cooler (8) together with the effluent from the dissociator still (3), yielding a solution containing 1.5% NH_3 which is returned to the H_2S washer.

EXAMPLE 2.

In place of removing the CO_2 and H_2S from the gas liquor and then distilling off the NH_3 to maintain the NH_3 concentration in the wash liquor, the gas liquor may be sprayed into the hot gas at the inlet of the condensers. The liquor removed in the condensers (2) (see figure 2) and leaving the final washer (4) is collected in tank (7) and then sprayed into the hot gas. The hydraulic main (1) liquor is fed to a fixed still together with lime, and the NH_3 evolved is also admitted to the gas at the inlet of the condensers. In this way the NH_3 content of the gas is increased to, say 430 grains per 100 cu. ft. The gas passes through the washer (3) where it is scrubbed with a 1% solution of NH_3. The NH_3 in the gas is reduced to 170 grains per 100 cu. ft. and the H_2S to 50 grains per 100 cu. ft. The wash liquor leaving the washer contains 1.4% NH_3 and 0.75% H_2S. This is fed to a dissociator still (5) where the H_2S and part of the HN_3 is evolved, the liquor leaving the still containing 1.0% NH_3. This, after cooling (6) is returned to the washer. The NH_3 and H_2S and any CO_2 are condensed and may be treated as in example 1. The excess H_2S may also be treated by any of the various methods proposed above.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the treatment of coal gas and other gases containing hydrogen sulphide and carbon dioxide, which consists in washing with a dilute solution of ammonium hydroxide in a washer under such conditions that the time of contact of the gases with the liquid is so short that hydrogen sulphide is selectively absorbed, without the addition of

ammonia from outside the system, characterised by the features that the ammonia from the liquor in the hydraulic main is used to supplement the ammonia in the absorption system and that only about 70—90% of the hydrogen sulphide is absorbed by keeping the ratio of H_2S to NH_3 in the wash liquor between about 1:1 and 1:1.5 parts by weight, while the balance of the hydrogen sulphide is removed by an iron oxide purifier so that the process is substantially self-sustaining.

2. A process as claimed in claim 1 in which the ammonia content of the gases is increased prior to the washing, by re-volatilising ammonia from the gas liquor produced in the normal purifying process into the gas stream, and the extra ammonia is removed simultaneously with the hydrogen sulphide by washing.

3. Process as claimed in claims 1 or 2, in which crude ammonia liquor from the hydraulic mains and condensers is dissociated in a dissociator, heated with lime and the ammonia evolved distilled off and used to regenerate the wash liquor.

4. A process as claimed in claims 1, 2 or 3 in which the ammonium sulphide solution is distilled so that substantially all the hydrogen sulphide but only half the ammonia is liberated these gases being absorbed to form a 4—5% solution of ammonium bisulphide, the whole of the ammonia and about 90% of the hydrogen sulphide being absorped.

5. A process as claimed in claims 1, 2 or 3 in which the ammonium sulphide solution is distilled so that all the hydrogen sulphide but only half of the ammonia is liberated and these gases are absorbed to form a 15—20% solution of ammonium sulphides; the whole of the ammonia and about 70% of the hydrogen sulphide being absorbed, and giving a solution in which the H_2S/NH_3 ratio is 1.5 by weight.

6. A process as claimed in claim 4 in which the hydrogen sulphide is burnt in excess of air to form sulphur dioxide which is absorbed in the original ammonia solution to form ammonium bisulphite.

Dated this 25th day of May, 1941.

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551,652 COMPLETE SPECIFICATION 1 SHEET

2nd Edition

[This Drawing is a reproduction of the Original on a reduced scale.]

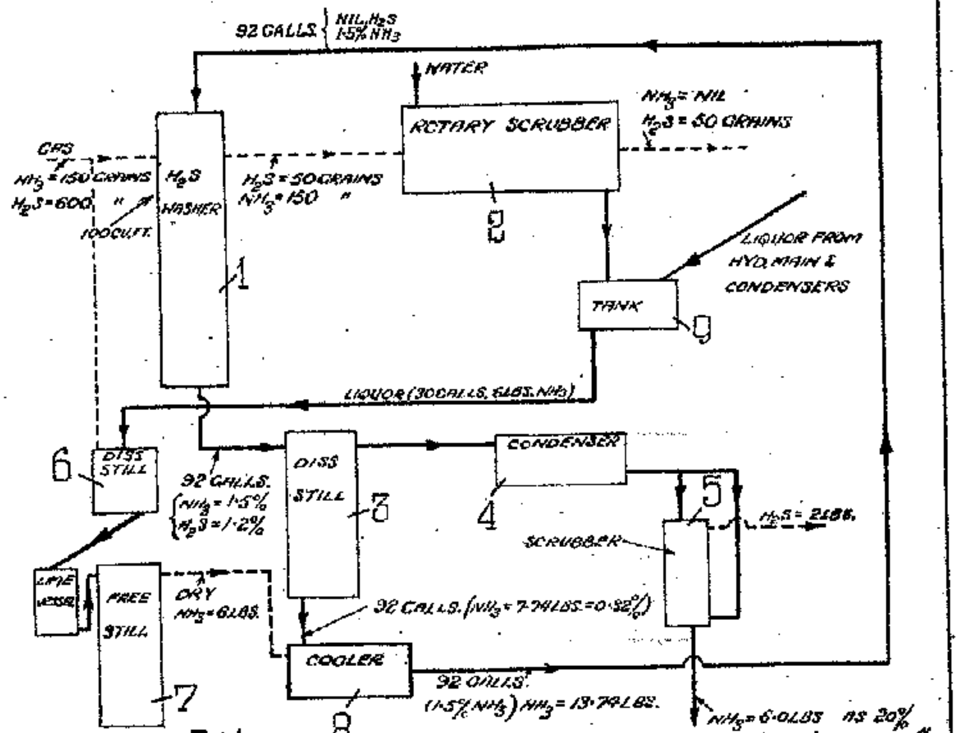

Fig. 1.

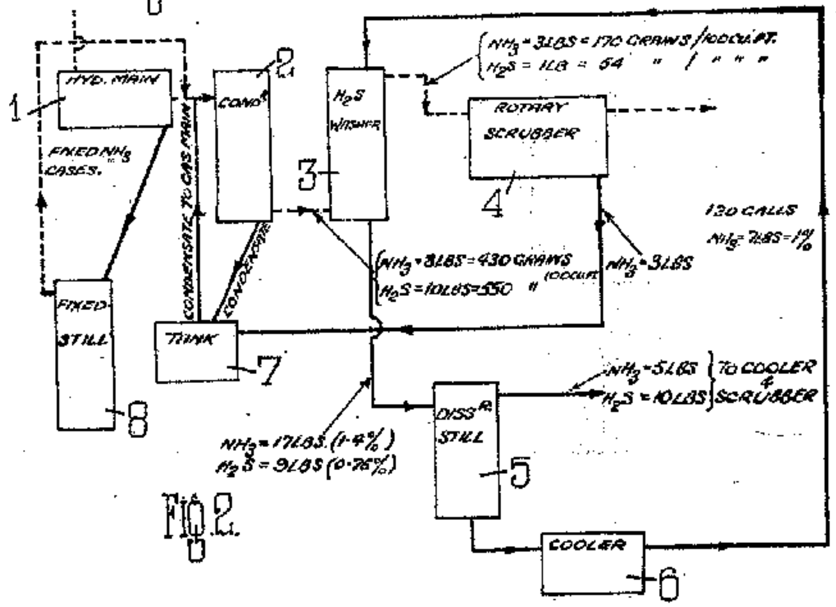

Fig. 2.

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