

Device and process for the condensation, separation and storage of liquid sulphur in a Claus plant

[0001] The invention relates to a device for the condensation, separation and storage of sulphur in a Claus plant, the Claus plant consisting of a Claus furnace, a waste heat boiler and a Claus reactor, and these plant sections being supported on a base or a comparable device, and an immersion chamber being provided at a level below the Claus plant and, if installed, also below an upstream device for gas scrubbing, the chamber taking up the sulphur in a siphon-like manner, the excess sulphur being able to flow from the immersion chamber into a vessel housing the immersion chamber. The invention also relates to a process with the aid of which liquid sulphur is fed to an immersion chamber, the immersion chamber being arranged at a height level below the waste heat boiler and the Claus reactor such that the liquid sulphur enters the immersion chamber without the aid of an additional conveying device.

[0002] The production of industrial gases frequently yields a sulphur-containing gas which must be freed from sulphur compounds. Treatment is typically carried out in a gas scrubber in which the sulphur-containing gas is brought into contact with an absorption liquid which is freed again from these sulphur compounds in a subsequent desorption step such that these can be passed to a Claus process. Typical industrial gases which must be freed from sulphur compounds are coke oven gas, natural gas or refinery gases.

[0003] The Claus process allows that the sulphur compounds are converted to elemental sulphur, this conversion being started by burning the sulphur compounds with the aid of an oxygen-containing gas. This gives elemental sulphur, whereas carbon and hydrogen react to form carbon dioxide and water. The combustion gas is fed to a waste heat boiler in which the sulphur condenses to a liquid. The liquid sulphur can then be passed to a further process step or disposal. The condensed and desulphurised gas from the waste heat boiler is fed to a Claus reactor where the residual sulphur compounds are passed to a combustion stage, sulphur dioxide being obtained which, by reacting with residual hydrogen sulphide in the presence of a catalyst, is converted to elemental sulphur. The reaction step for the conversion of sulphur dioxide with hydrogen sulphide can be provided several times to achieve the maximum degree of desulphurisation.

[0004] A typical embodiment of the Claus process is disclosed in EP 1527013 B1. This teaching lays claim to a process for the separation of hydrogen sulphide from coke oven gas, in which the hydrogen sulphide is removed from the coke oven gas by gas scrubbing using an absorption liquid, the laden absorption liquid is regenerated and the laden absorption liq-

uid and the hydrogen sulphide obtained in concentrated form are fed to the Claus plant, the hydrogen sulphide reacting with atmospheric oxygen in a Claus boiler of the Claus plant giving elemental sulphur, and the process gas leaving the Claus boiler being cooled down in a waste heat boiler to a temperature required for the condensation of the sulphur, then, after
 5 separation of the sulphur, being heated and fed to a reaction furnace of the Claus plant. In said reactor the sulphur compounds react in the presence of a catalyst to give elemental sulphur, and the process gas leaving the reaction furnace is cooled down to a temperature required for the condensation of the sulphur, and the condensed sulphur is separated, the Claus plant according to the invention being operated by one single reaction furnace only,
 10 the operating temperature of which being adjusted to less than 250°C, and the process gas leaving the reaction furnace is recycled after separation of the condensed sulphur to the coke oven gas to be treated together with residual hydrogen sulphide not converted in the reaction furnace prior to gas scrubbing. The teaching does not refer to any storage and further processing of the sulphur obtained in the process.

15 **[0005]** Normally, this sulphur is separated in liquid form and stored after solidification. The solid sulphur can be further used for versatile purposes, for example, as starting material for chemical syntheses or for the production of sulphuric acid. In the condensation of the sulphur, part of the sulphur is obtained in liquid form, whereas the remainder is still contained in the form of fine droplets in the condensed gas. To facilitate the formation of sulphur droplets
 20 of a size suitable for separation, the condensed gas can be fed to a siphon-type device where an almost complete separation of sulphur is achieved.

[0006] DE 2756992 C2 gives an example of a device which facilitates complete condensation of the sulphur contained in a sulphur-containing combustion gas. The said teaching discloses a device for the separation of sulphur droplets from a gas stream, preferably from a
 25 reaction gas stream of a Claus catalyst, this device consisting of a housing, a tube bundle arranged in the housing between the tubesheets, nozzles on the housing for the supply and discharge of a coolant flowing around the tube bundle and one gas stream inlet and outlet nozzle each on the housing on the inflow and outflow side of the tube bundle, with a separating unit, passed by the gas stream, being arranged in the housing between the tubesheet on
 30 the gas outlet side of the tube bundle and the gas outlet nozzle as well as outlets on the housing bottom on the inflow and outflow side of the separating unit for the sulphur separated upstream or in and downstream of the separating unit according to the invention. In an embodiment the device includes lines which convey the liquid sulphur from a separating unit to a sulphur collecting tank where they dip-in in a siphon-like manner.

[0007] The embodiment mentioned has the disadvantage that the sulphur withdrawn from the discharge nozzle cannot be stored but must be transported onwards in liquid form. In addition, no supply option for liquid sulphur as obtained, for example, in a waste heat boiler of a Claus plant is provided.

5 **[0008]** It is therefore the objective to provide a device which facilitates condensation of sulphur from a sulphur-containing gas, in which fine sulphur droplets contained in the gas should also condense, and a supply option for liquid sulphur already condensed from a waste heat boiler should be given. The device should also allow the storage of the sulphur.

10 **[0009]** The invention achieves the objective by a device consisting of a siphon-type immersion chamber arranged in a vessel, this vessel being arranged on a lower level than the other plant sections of the Claus plant such that the sulphur can enter the immersion chamber without the aid of an additional conveying device, and the immersion chamber being designed in a siphon-like manner such that the gas is immersed into the liquid in the vessel.

15 **[0010]** Via the outlet means arranged downstream of the siphon, the liquid sulphur flows from the immersion chamber to the vessel from where it can be transported onwards.

[0011] Claim is particularly laid to a device for the condensation, separation and storage of liquid sulphur, comprising

- 20 • a gas scrubbing unit by means of which the hydrogen sulphide is removed from the coke oven gas by gas scrubbing using an absorption liquid, the laden absorption liquid is regenerated and the hydrogen sulphide obtained in concentrated form is fed to the Claus plant consisting of Claus furnace, waste heat boiler and Claus reactor, and
- a Claus furnace in which the sulphur compounds are burnt with atmospheric oxygen to give elemental sulphur,
- 25 • a waste heat boiler in which the process gas leaving the Claus furnace is cooled to a temperature required for the condensation of the sulphur, is reheated after separation of the sulphur and fed to a Claus reactor,
- a Claus reactor in which part of the residual hydrogen sulphide is burnt, and the forming sulphur dioxide is converted to sulphur by reacting with residual
- 30 • hydrogen sulphide in the presence of a catalyst,
- a condensation chamber related to the Claus reactor,

and which is characterised in that

- the Claus plant and, if required, also the gas scrubbing unit are supported on a base or a suitable device, and a siphon-type immersion chamber is arranged below such device in a heatable, ground-level vessel, and
- the Claus plant and, if required, also the gas scrubbing unit are located at a higher level than the vessel and the immersion chamber contained therein such that no conveying equipment is required for conveying the sulphur to the immersion chamber, with
- the Claus plant and, if required, also the gas scrubbing unit being located at an elevated level of at least 4.00 metres above the lowest discharge point for sulphur in the immersion chamber.

[0012] The vessel is arranged at ground level, which is to be understood as an arrangement at approximated ground level. A deep pit for lowering the vessel to a notable extent, as frequently found in Claus plants for the storage of sulphur, incurring the related costs, is not required.

[0013] In principle, the vessel and the immersion chamber can be made of any material desired. In a preferred embodiment the vessel and the immersion chamber are made of steel in order to be adequately corrosion-resistant to the temperatures of the liquid sulphur. The vessel can be heated to ensure that the sulphur contained therein is constantly kept liquid. The immersion chamber as well can be heated or thermally insulated.

[0014] In order to operate the immersion chamber in an optimum way the sulphur must also be liquid even in the immersion chamber such that the siphon can work properly. During operation this can be accomplished by supplying an adequate amount of sulphur condensate. For this purpose, the immersion chamber can also be provided with heat-insulated wall materials. If an adequate supply of liquid sulphur is not always ensured, the immersion chamber can also be equipped with a device for heating the sulphur in order to prevent the sulphur from solidifying in the immersion chamber.

[0015] Collecting or storage tanks can be allocated to the Claus plant or the vessel housing the immersion chamber, these tanks being piped to the vessel or the immersion chamber. In a preferred embodiment, the storage or collecting tank(s) as well is/are made of steel. The type of arrangement also allows for the saving of a considerable space volume.

[0016] Immersion cylinders which prevent an undesired escape of desulphurised gas in Claus plants according to prior art are no longer required. The installation of safety immer-

sion cylinders, for example, for process gas on the burner, is always feasible according to the invention.

[0017] In an embodiment of the device the Claus plant is equipped with several Claus reactors. This corresponds to embodiments commonly used in prior art. However, in a further embodiment it is also possible to provide the Claus reactor only once as disclosed in an exemplary manner in EP 1527013 B1. Finally, the inventive device can also include pumps or conveying equipment for the liquid sulphur. The device or plant can, for example, also include blowers, separators, silencers, storage tanks or degasifiers. Of course, the vessel can also be equipped with suspension devices for dispatch.

[0018] To allow installation of the Claus plant at a higher level than the immersion chamber and the vessel, the Claus plant is supported, for example, on a platform or a base. This can be a grid tray, for example, to be supported on stilts.

[0019] Claim is also laid to a process for the condensation, separation and storage of liquid sulphur from a Claus process. Claim is particularly laid to a process for the condensation, separation and storage of liquid sulphur, in which

- the hydrogen sulphide is removed from the coke oven gas by gas scrubbing using an absorption liquid, the laden absorption liquid is regenerated and the hydrogen sulphide obtained in concentrated form is fed to the Claus plant, and
- the hydrogen sulphide is converted to sulphur in a Claus furnace by reacting with an oxygen-containing gas, and the combustion gas is fed to a waste heat boiler in which the liquid sulphur condenses, and
- the cooled process gas is fed to a Claus reactor in which part of the residual hydrogen sulphide is burnt to form sulphur dioxide, and the sulphur dioxide is converted to sulphur by reacting with hydrogen sulphide in the Claus reactor,

and which is characterised in that

- the sulphur is fed in a siphon manner from the waste heat boiler and the Claus reactor to an immersion chamber housed in a vessel located at a lower level, the said vessel being located at ground level below the level of the entire Claus plant, and
- the immersion chamber is housed in a vessel taking up the liquid sulphur from the immersion chamber,

- with the flowing sulphur overcoming a difference in altitude of at least 4.00 metres by gravity.

[0020] In this context hydrogen sulphide refers to a gas or a fraction which contains hydrogen sulphide as the main constituent. This fraction may also contain organic sulphur compounds. In a preferred embodiment the sulphur is fed to the immersion chamber in liquid form and kept in the vessel at a temperature between 110°C and 300°C. If there is an adequate flow of sulphur, the latter remains liquid of its own accord. It is preferably also possible to keep the sulphur at a temperature between 110°C and 300°C by heating it in the immersion chamber. This can be done by heating elements attached to the immersion chamber.

[0021] The inventive device and the inventive process have the following advantages: Conveying the liquid sulphur from the Claus plant to the storage facility is not required because the pipes are short on account of the difference in altitude to be overcome; the sulphur can be stored in the downstream heatable vessel even if no sulphur is temporarily taken from the plant such that a separate storage tank is not required. A deep pit is not required; the immersion cylinders which prevent the escape of desulphurised gas in Claus plants according to prior art are no longer required.

[0022] The invention is explained by a drawing, which represents only an exemplary embodiment of the invention. A sulphur-containing concentrate (3) is recovered from a coke oven gas (1, COG) by means of a gas scrubbing process (2) and burnt with atmospheric oxygen to give elemental sulphur, water and carbon dioxide in a Claus furnace (4). One obtains elemental sulphur (5) in gaseous form. This is fed to a waste heat boiler (6). In this, a tail gas (7) and liquid sulphur (8) are obtained by condensation, the tail gas (7) still containing sulphurous gases. These are completely burnt in the combustion stage (9) of a Claus reactor. One obtains sulphur dioxide (10). This is converted to form elemental sulphur in the catalyst stage (11) of a Claus reactor with condensation chamber by reacting with supplied hydrogen sulphide from the gas scrubbing process. One obtains a desulphurised combustion gas (12) or off-gas. This stage as well yields elemental sulphur (13) in liquid form. All plant sections (2 to 11) are supported on a base (14) which is, for example, a grid tray supported on stilts (15) at a higher level. The liquid sulphur from the waste heat boiler (8) and from the catalyst stage (11) is fed via pipes to an immersion chamber (16) equipped with a partition wall (17). As a result, this acts as a siphon. In this way, the total sulphur condenses. The sulphur is kept liquid by continued flowing and by heating and fed to a vessel (19) via a discharge nozzle (18). There it is stored in liquid or solid form depending on the demand.

[0023] List of reference numbers and designations

- 1 Coke oven gas
- 2 Gas scrubber
- 3 Sulphur-containing concentrate
- 4 Claus furnace
- 5 Elemental gaseous sulphur
- 6 Waste heat boiler
- 7 Sulphur-containing tail gas
- 8 Liquid elemental sulphur from the waste heat boiler
- 9 Combustion stage of the Claus reactor
- 10 Sulphur dioxide
- 11 Catalyst stage of the Claus reactor with condensation chamber
- 12 Desulphurised combustion gas
- 13 Liquid elemental sulphur from the Claus reactor
- 14 Base
- 15 Stilts
- 16 Immersion chamber
- 17 Partition wall
- 18 Discharge nozzle
- 19 Vessel

Claims

1. Device for the condensation, separation and storage of liquid sulphur, comprising

- a gas scrubbing (2) unit by means of which the hydrogen sulphide is removed from the coke oven gas by gas scrubbing (2) using an absorption liquid, the laden absorption liquid is regenerated and the hydrogen sulphide obtained in concentrated form is fed to the Claus plant consisting of Claus furnace (4), waste heat boiler (6) and Claus reactor (9), and
- a Claus furnace (4) in which the sulphur compounds are burnt with atmospheric oxygen to give elemental sulphur,
- a waste heat boiler (6) in which the process gas leaving the Claus furnace (4) is cooled to a temperature required for the condensation of the sulphur, is reheated after separation of the sulphur and fed to a Claus reactor (9),
- a Claus reactor (9) in which part of the residual hydrogen sulphide is burnt, and the forming sulphur dioxide is converted to sulphur by reacting with residual hydrogen sulphide in the presence of a catalyst,
- a condensation chamber (11) related to the Claus reactor (9),

characterised in that

- the Claus plant and, if required, also the gas scrubbing (2) unit are supported on a base (14) or a suitable device, and a siphon-type immersion chamber (16) is arranged below such device (14) in a heatable, ground-level vessel (19), and
- the Claus plant and, if required, also the gas scrubbing (2) unit are located at a higher level than the vessel (19) and the immersion chamber (16) contained therein such that no conveying equipment is required for conveying the sulphur to the immersion chamber (16),
- with the Claus plant and, if required, also the gas scrubbing (2) unit being located at an elevated level of at least 4.00 metres above the lowest discharge point for sulphur in the immersion chamber (16).

2. Device for the condensation, separation and storage of liquid sulphur according to claim 1, **characterised in that** the vessel (19) and the immersion chamber (16) are made of steel.

3. Device for the condensation, separation and storage of liquid sulphur according to claim 1 or 2, **characterised in that** the immersion chamber (16) as well is equipped with a device for heating of sulphur.

4. Device for the condensation, separation and storage of liquid sulphur according to any of claims 1 to 3, **characterised in that** collecting or storage tanks are allocated to the Claus plant or the vessel (19) housing the immersion chamber (16), these tanks being piped to the vessel.

5. Device according to claim 4, **characterised in that** the storage or collecting tank(s) is/are made of steel.

6. Device for the condensation, separation and storage of liquid sulphur according to any of claims 1 to 5, **characterised in that** there are several Claus reactors.

7. Process for the condensation, separation and storage of liquid sulphur, in which

- the hydrogen sulphide is removed from the coke oven gas by gas scrubbing (2) using an absorption liquid, the laden absorption liquid is regenerated and the hydrogen sulphide obtained in concentrated form is fed to the Claus plant, and
- the hydrogen sulphide is converted to sulphur in a Claus furnace (4) by reacting with an oxygen-containing gas, and the combustion gas is fed to a waste heat boiler (6) in which the liquid sulphur condenses, and
- the cooled process gas is fed to a Claus reactor (9) in which part of the residual hydrogen sulphide is burnt to form sulphur dioxide, and the sulphur dioxide is converted to sulphur by reacting with hydrogen sulphide in the Claus reactor (9),

characterised in that

- the sulphur is fed in a siphon manner from the waste heat boiler (6) and the Claus reactor (9) to an immersion chamber (16) housed in a vessel located at a lower level, the said vessel (19) being located at ground level below the level of the entire Claus plant, and
- the immersion chamber (16) is housed in a vessel (19) taking up the liquid sulphur from the immersion chamber (16),

- with the flowing sulphur overcoming a difference in altitude of at least 4.00 metres by gravity.

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8. Process for the condensation, separation and storage of liquid sulphur according to claim 7, **characterised in that** the sulphur is fed to the immersion chamber (16) at a temperature between 110°C and 300°C.
9. Process for the condensation, separation and storage of liquid sulphur according to claim 7 or 8, **characterised in that** the sulphur is kept at a temperature between 110°C and 300°C by heating it in the immersion chamber (16) and the vessel (19).