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(54) Titre : PROCEDE ET DISPOSITIFS POUR PRODUIRE DE LA NAPHTALINE A PARTIR DE GAZ BRUT DE
COKERIE

(54) Title: METHOD AND DEVICES FOR PRODUCING NAPHTHALENE FROM COKE PRODUCED FROM COKE
OVEN CRUDE GAS

(57) **Abrégé/Abstract:**

The invention relates to a method for producing naphthalene from coke oven crude gas. The coke oven crude gas is directly cooled by means of sprinkling water thereon, and the naphthalene is obtained by subsequent refrigerating and treatment. The invention is characterised in that after direct cooling, the coke oven crude gas is then guided through an electrofilter to be subsequently cooled in such a manner that the naphthalene contained in the coke oven crude gas is separated from the gaseous phase and is obtained in the form of pure naphthalene without any additional treatment.

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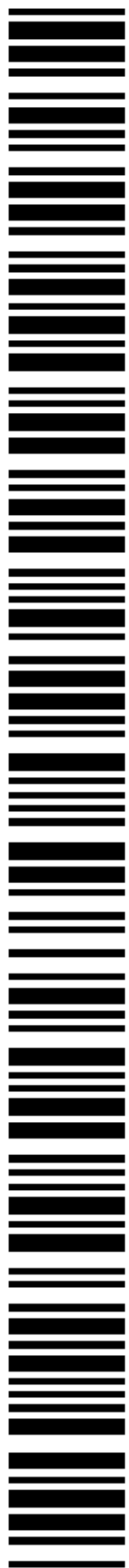
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(54) Title: METHOD AND DEVICES FOR PRODUCING NAPHTHALENE FROM COKE PRODUCED FROM COKE OVEN
CRUDE GAS

(54) Bezeichnung: VERFAHREN UND VORRICHTUNGEN ZUR GEWINNUNG VON NAPHTHALIN AUS KOKSOFFENROH-
GAS

(57) Abstract: The invention relates to a method for producing naphthalene from coke oven crude gas. The coke oven crude gas is directly cooled by means of sprinkling water thereon, and the naphthalene is obtained by subsequent refrigerating and treatment. The invention is characterised in that after direct cooling, the coke oven crude gas is then guided through an electrofilter to be subsequently cooled in such a manner that the naphthalene contained in the coke oven crude gas is separated from the gaseous phase and is obtained in the form of pure naphthalene without any additional treatment.

(57) Zusammenfassung: Verfahren zur Gewinnung von Naphthalin aus Koksoffenrohgas, bei dem das Koksoffenrohgas mittels Berieselungswasser direkt gekühlt und durch anschließende Kühlung und Aufarbeitung das Naphthalin gewonnen wird, wobei das Koksoffenrohgas nach der direkten Koksoffenrohgaskühlung durch einen Elektrofilter geleitet wird und anschließend derart gekühlt wird, dass das in dem Koksoffenrohgas enthaltene Naphthalin aus der Gasphase abgeschieden und ohne weitere Aufarbeitung als Rein-Naphthalin gewonnen wird.



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TRANSLATION

METHOD AND APPARATUS FOR RECOVERING NAPHTHALENE
FROM COKE OVEN CRUDE GAS

The invention relates to a method of recovering
5 naphthalene from coke oven crude gas according to the preamble of
claim 1 as well as apparatus suitable for that purpose.

In the thermal treatment of coal under the exclusion of
air in a coke oven, coke oven crude gas is produced which is cooled
by means of water droplets [trickling water] in a crude gas
10 condenser to about 80°C and is saturated with water vapor. As a
result, about 20% of the naphthalene entrained with the coke oven
crude gas is bound up in tar in the liquid phase. The remaining
80% of the naphthalene is then condensed in an indirect crude
gascooling together with tarry aqueous components in a precooler.
15 This condensate is initially fed to the crude gas condenser, there
heated to about 80°C and supplied to a tar separation in which the
separation of crude tar from cooled water is effected. The
naphthalene contained in the crude tar is recovered from the crude
tar in highly expensive processes from a process technology view
20 point. Initially in a distillation of the tar a naphthalinol is
produced is then subjected to crystallization in which the crude
naphthalene is deposited out to recover pure naphthalene, further
processes like chemical reactions, (for example with sodium
hydroxide and sulfuric acid) are required or the raw naphthalene is
25 distilled again.

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The invention has as its object to provide a method of recovery of naphthalene from coke oven crude gas as well as to provide appropriate apparatus by which the naphthalene can be recovered in high purity at low cost in a simple manner and way.

5 These objects are achieved with the features of claim 1.

Further details are found in the features of the dependent claims.

10 According to the invention, the crude gas coming from the coke oven is cooled as is customary with the aid of sprinkling water thereon in the crude gas condenser directly to about 80°C. This results in a liquid phase and a gas phase. The liquid phase is fed to tar separation in which the separation into tar and coal water is effected. A part of the coal water is again used for the direct crude gas cooling as the trickling water. The gaseous phase
15 is fed through an electrostatic filter in which the solids content and tar content of the coke oven crude gas is reduced at a temperature of about 80°C by 99.99%.

 Since the deposition in the electrostatic filter is carried out at a temperature of about 80°C, the naphthalene
20 (predominantly in the gaseous form) remains in the coke oven crude gas after passing the electrostatic filter. The coke on crude gas is then cooled from about 80°C to about 20°C such that the naphthalene contained in the crude gas deposits out. Since the solids as well as the tar has already been deposited in the
25 electrostatic filter, the naphthalene here is a very pure

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naphthalene which is a saleable product which can be delivered directly to the consumer.

5 So that the naphthalene in the electrostatic filter will not separate out, the electrostatic filter is isolated or even heated so that the isothermal commands in the electrostatic filter will be maintained at about 80°C. The tar which has been separated out [in the electrostatic filter] according to the invention serves to protect the inner surfaces of the electrostatic filter from corrosion, the electrostatic filter can be composed of simple
10 structural steel (for example: ST 37-3).

The tar deposited in the electrostatic filter is especially low in naphthalene since, as has been observed above, the naphthalene remains in the coke oven gas. This low naphthalene tar is supplied to a tar separation stage. Based upon the low
15 naphthalene content (about 2% residual naphthalene content), emulsion formation in the tar-water separation is reduced and the tar separation improved. In addition, the residence time required for the tar-water separation is also reduced. The tar water separation can also be carried out at a somewhat lower temperature
20 since the emulsion formation is reduced.

According to a variant of the process, it is also possible to feed the low naphthalene tar from the electrostatic filter, since it is close to water free, not to the tar separation stage but directly to the tar storage receptacle. This relieves
25 load on the tar separation stage. The low naphthalene tar can also

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be stored in a separate tar storage receptacle and used for other processes like, for example, precooler flushing.

The naphthalene can be recovered from the crude gas downstream of the electrostatic filter by direct or indirect
5 cooling.

In the direct cooling, the coke oven crude gas is directly cooled with water downstream of the electrostatic filter. The naphthalene is thereby separated out and is then separated from the water. Since the water is approximately free from impurities,
10 it can be fed in a circulation with corresponding recooling. The recooling of the circulated water can be effected directly or indirectly. The excess water is supplied to the condenser.

With indirect cooling the naphthalene crystallizes on the cooling surfaces.

15 The naphthalene which is crystallized out can be recovered by different techniques and in various ways: One possibility is to recover the naphthalene by heating. The heating can be effected directly or indirectly. The cooling surfaces can be heated by a heating medium, both on the "naphthalene side" and
20 also upon the other side and the naphthalene thus removed and transported through heated conduits to the shipment point.

The heating can be effected also by heating rods which can be built into the apparatus at appropriate locations.

Another possibility is to flush the naphthalene with
25 previously heated liquid naphthalene. Liquid naphthalene can also be used for the indirect heating.

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It is also possible to dissolve the crystallized-out naphthalene with a solvent from the cooling surfaces and then to recover it from the solvent.

5 The recovery of the crystallized-out naphthalene can also be effected mechanically, for example, with the aid of a shaver. Either the shaver or the cooling surface is moved to recover the naphthalene.

10 It is also possible to recover the crystallized-out naphthalene with the aid of mechanical vibrations which can be produced by an impactor or shaker or with the aid of an imbalanced motor.

15 In the aforescribed method the recovery of naphthalene is carried out discontinuously, that is in a first method step the naphthalene is crystallized out and in a second method step the crystallized-out naphthalene is recovered.

20 It is however also possible to carry out the recovery of naphthalene continuously or quasicontinuously. In this mode of operation, a device is fed segment wise into the cooling region or into the naphthalene recovery region. A continuous operation is also possible with the use of a shaver or vibration generator.

For carrying out the method of the invention, it is only necessary to have an electrostatic filter and a device appropriate for the deposition of naphthalene from which the deposited can be recovered.

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For an after-fitting of existing coking plants it is possible to provide only the electrostatic filter and use the previously provided precooler.

5 If the electrostatic filter is provided downstream from the precooler, the electrostatic filter can be built into the crude gas path upstream of the precooler.

10 This electrostatic filter can be built into the crude gas path ahead of the precooler. Electrostatic filters following the precooling are superfluous in the method according to the invention.

Since existing coke plants as a rule have multiple precoolers, the method can be carried out as follows with the precoolers which have previously been provided:

15 One group of for example two precoolers is used to crystallize out the naphthalene. The remaining precoolers run in the usual precooler operation with tar flushing. In a quasi continuous operation, for example, one precooler is used to crystallize out the naphthalene and one precooler is used for the naphthalene recovery. With an increase in the pressure loss in the
20 precooler for naphthalene recovery or with an increase in the crude gas discharge temperature by a maximum of 2°C, the crude gas is no longer fed through this precooler but through the [other] precooler which is in standby status following the naphthalene recovery.

25 The naphthalene crystallized out on the cooler tubing is recovered by heating the tubing up. The cooler tubing can be heated from the interior or from the exterior. A combination of

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internal and external heating is however also possible. After the naphthalene has been recovered from the precooler, it is switched over and the precooler can again be used for crystallizing out of the naphthalene while the naphthalene is recovered from the other
5 precooler.

In this mode of operation, a reduced naphthalene recovery can be carried out and a switchover to partial or also complete cooler operation in the classical manner.

10 In this mode of precooler operation it is advantageous to prevent the naphthalene from plugging up the precooler by flushing the precooler with the low naphthalene tar from the electrostatic filter.

15 With the method according to the invention of recovering naphthalene from coke oven crude gas, the apparatus suitable for recovering naphthalene can use a conventional precooler. In the cooling step, the naphthalene crystallizes out on the cooling tubing from the gas phase and then the naphthalene is recovered in accordance with the above described process. During the naphthalene recovery the coke oven crude gas is lead through
20 the precooler which is in a stand by state.

The precooler can be equipped for a quasicontinuous naphthalene recovery. Then the precooler must be equipped segment wise with controllable valves. In this manner and way it is possible to carry out simultaneously in the precooler a cooling
25 operation for crystallizing out the naphthalene and a naphthalene recovery operation with heating up of the naphthalene.

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For the recovery of the naphthalene, other apparatus is also possible. It is only necessary that a cooling surface be provided from, which using the above described process, the naphthalene can be recovered. As cooling surfaces for example, a
5 drum or disk can be used. If this drum or these disks are equipped with a shaver, the naphthalene recovery can also be effected continuously.

It is also possible to crystallize out the naphthalene in a tube cooler whose tubes are cooled at their outer walls. The
10 naphthalene then deposits on the tubing inner walls and can be recovered with the described method.

The aforescribed and claimed components and those used as described in the examples according to the invention can be selected as to their dimensions, their shapes, their choice of
15 materials and their technological conceptions, without special preconditions so that they can be employed without limitations in the full range of applications.

Further details, features and advantages of the subject of the invention are given in the dependent claims and in the
20 following description relevant to the accompanying drawing in which preferred embodiments of the method of the invention are given by way of example and are illustrated.

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The drawing shows:

FIG. 1 is a schematic illustration of the method for recovering naphthalene from coke oven crude gas with direct cooling;

5 FIG. 2 is a schematic illustration of the method for recovering naphthalene from coke oven crude gas with indirect cooling; and

10 FIG. 3 is a schematic illustration of the method variant according to which part from the recovery of naphthalene a conventional crude gas filling can be carried out.

From FIG. 1 it is seen that a crude gas coming from the coke oven 1 is cooled in a crude gas condenser 2 with water droplets supplied by a line 10 from a temperature of about 800°C to about 80°C. The coke oven crude gas is fed through an
15 electrostatic filter three in which the solids and tar content of the coke oven crude gas is reduced by 99.99%. The precipitated tar is fed via a line 7 to the tar separation or to the tar collecting vessel. The coke oven crude gas is fed to a device 4 for separating out the naphthalene in which the naphthalene is
20 separated out by direct cooling with water and is separated from the water. The naphthalene is fed via a line 8 to a separating device which has not been shown and in which the naphthalene is separated from residual water adhering to it. This residual water can be supplied as cooling water. The cooling water is fed in a
25 circulating path through a line 11, a cooler 12 and a line 13.

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Surplus water is fed via a line 9 to the crude gas condenser 2. The coke oven crude gas is fed to a cleaning unit 5 where it is freed from hydrogen sulfide, ammonia and optionally from benzene before it is supplied to a coke oven gas consumer 6.

5 In FIG. 2, the process variant with indirect cooling for the recovery of naphthalene has been illustrated. The reference characters have the same significance as in FIG. 1. The coke oven crude gas is fed from the electrostatic filter 3 into a device 4 for crystallizing out the naphthalene and in which the
10 naphthalene is deposited on the cooling surface while the water vapor which is condensed out is carried away via the line 9 to the crude gas condenser 2. The naphthalene crystallized out on the cooling surfaces is drawn off as liquid naphthalene from the device 4 upon heating of the cooling surfaces and is transported through a
15 heated duct 8 to a shipment facility (not shown).

In FIG. 3, a process scheme has been shown in which two precoolers 4 and 4' are used for crystallizing out the naphthalene. The device 4 is used for naphthalene recovery, that is obtaining the pure naphthalene as transported by the line 8 to the shipment
20 facility. The device 4' is used in precooler operation. The low-naphthalene tar is used in this case for flushing the precooler 4' to prevent blockage by naphthalene. The remaining reference characters have the same significance as in FIG. 1.

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Reference Characters

- | | | |
|----|-----|--|
| | 1. | Coke Oven |
| | 2. | Condenser |
| | 3. | Electrostatic Filter |
| 5 | 4. | Apparatus for cooling |
| | 5. | Coke Oven Gas Cleaning |
| | 6. | Coke Oven consumer |
| | 7. | Line [Conduit] (Tar) |
| | 8. | Line [Conduit] (Naphthalene) |
| 10 | 9. | Line [Conduit] (Condensate) |
| | 10. | Line [Conduit] (Trickling Water) |
| | 11. | Line [Conduit] (Cooling Water) |
| | 12. | Cooler |
| | 13. | Line [Conduit] (Cooling Water Recycle) |

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Patent Claims:

1. A method of recovering naphthalene from coke oven crude gas in which the crude gas is directly coupled by means of trickling water and through subsequent cooling and processing the naphthalene is recovered, characterized in that the coke oven crude gas, after the direct coke oven crude gas cooling, is conducted through an electrostatic filter and then so cooled that the naphthalene contained in the coke oven crude gas is precipitated from the gas phase and without further processing is recovered as pure naphthalene.

2. The method according to claim 1 characterized in that the electrostatic filter is so insulated or heated that it operates isothermally.

3. The method according to claims 1 and 2 characterized in that the cooling of the coke oven crude gas following the electrostatic filter is a direct cooling.

4. The method according to claims 1 and 2 characterized in that the cooling of the coke oven crude gas following the electrostatic filter is an indirect cooling.

5. The method according to claims 1 to 4 characterized in that the separated naphthalene is recoupled by heating up.

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6. The method according to claim 5 characterized in that the heating up is effected indirectly.

7. The method according to claim 5 characterized in that the heating up is effected indirectly.

5 8. The method according to claims 6 and 7 characterized in that the heating up is carried out with already heated liquid naphthalene.

9. The method according to claims 1 and 2 characterized in that the deposited naphthalene is dissolved with a solvent and
10 the solvent is then removed.

10. The method according to claim 1 characterized in that the separated out naphthalene is mechanically recovered.

11. The method according to claims 1 and 2 characterized in that the separated out naphthalene is continuously recovered.

15 12. An apparatus for carrying out the method according to claim 1 characterized in that a conventional precooler is used for separating out the naphthalene.

13. The apparatus according to claim 12 characterized in that heating rods are provided in the precooler.

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14. The apparatus for carrying out the method according to claim 1 characterized in that a precooler is equipped with controllable valves in a segmentwise manner.

5 15. An apparatus for carrying out the method according to claim 1 characterized in that a cooling surface is provided with at least one shaver.

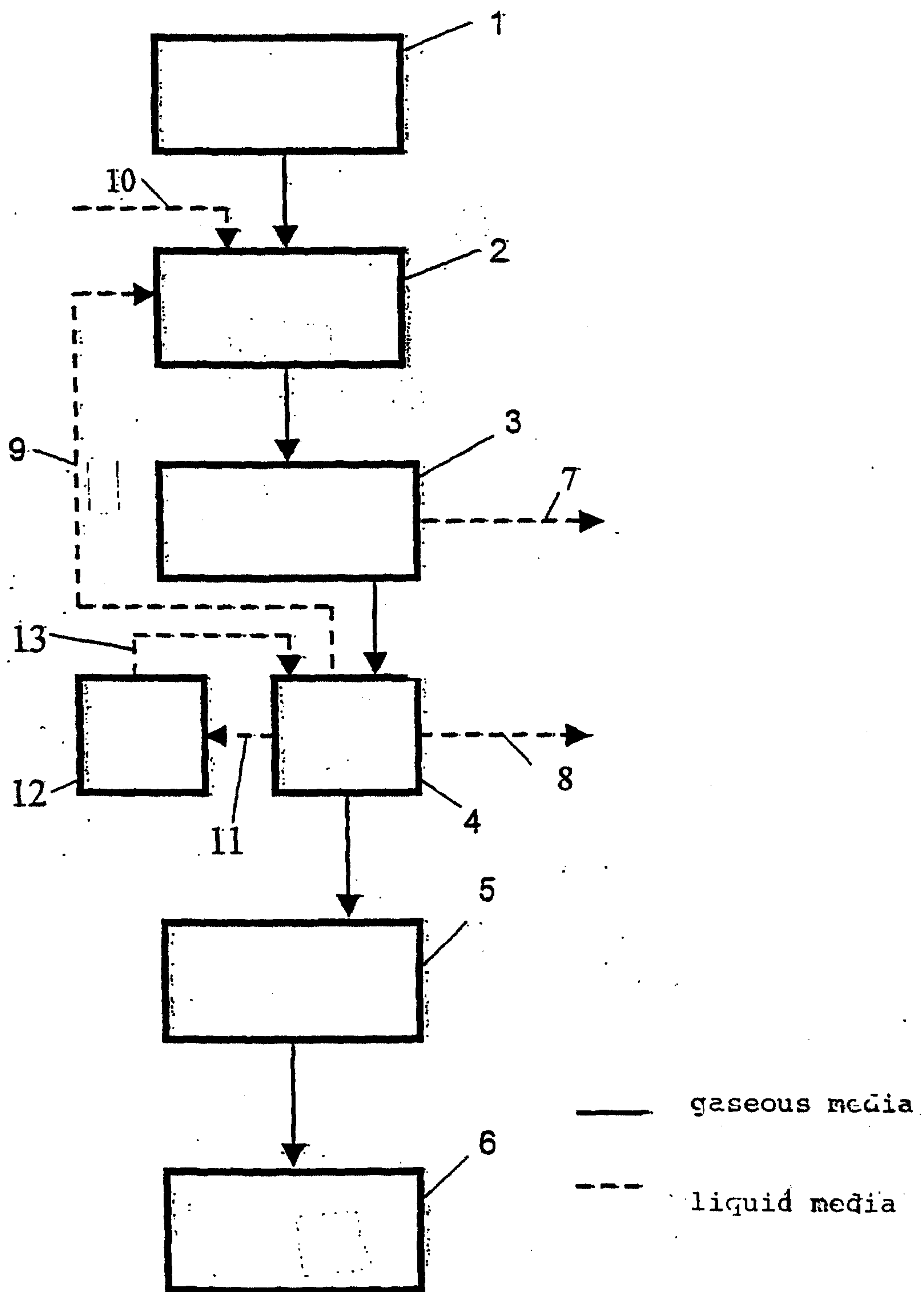
16. The apparatus for carrying out the method according to claim 1 characterized in that a cooled drum equipped with shavers is provided.

10 17. An apparatus for carrying out the method according to claim 1 characterized in that a cooled disk equipped with shavers is provided.

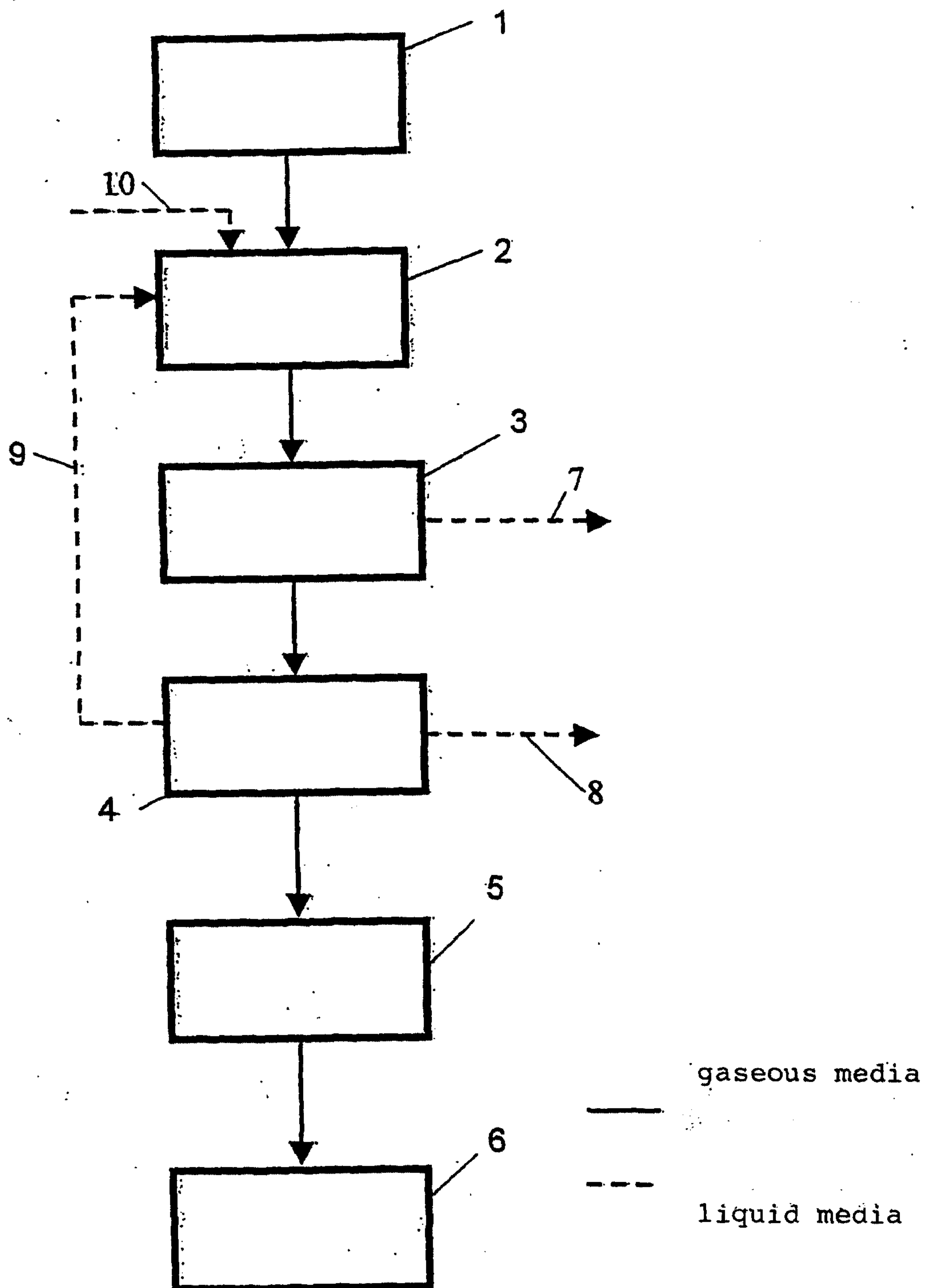
15 18. An apparatus for carrying out the method according to claim 1 characterized in that a tube cooler is provided with cooling of the outer walls of the tubes.

19. An apparatus for carrying out the method according to claim 1 characterized in that at least one vibration generator is provided at a cooling surface.

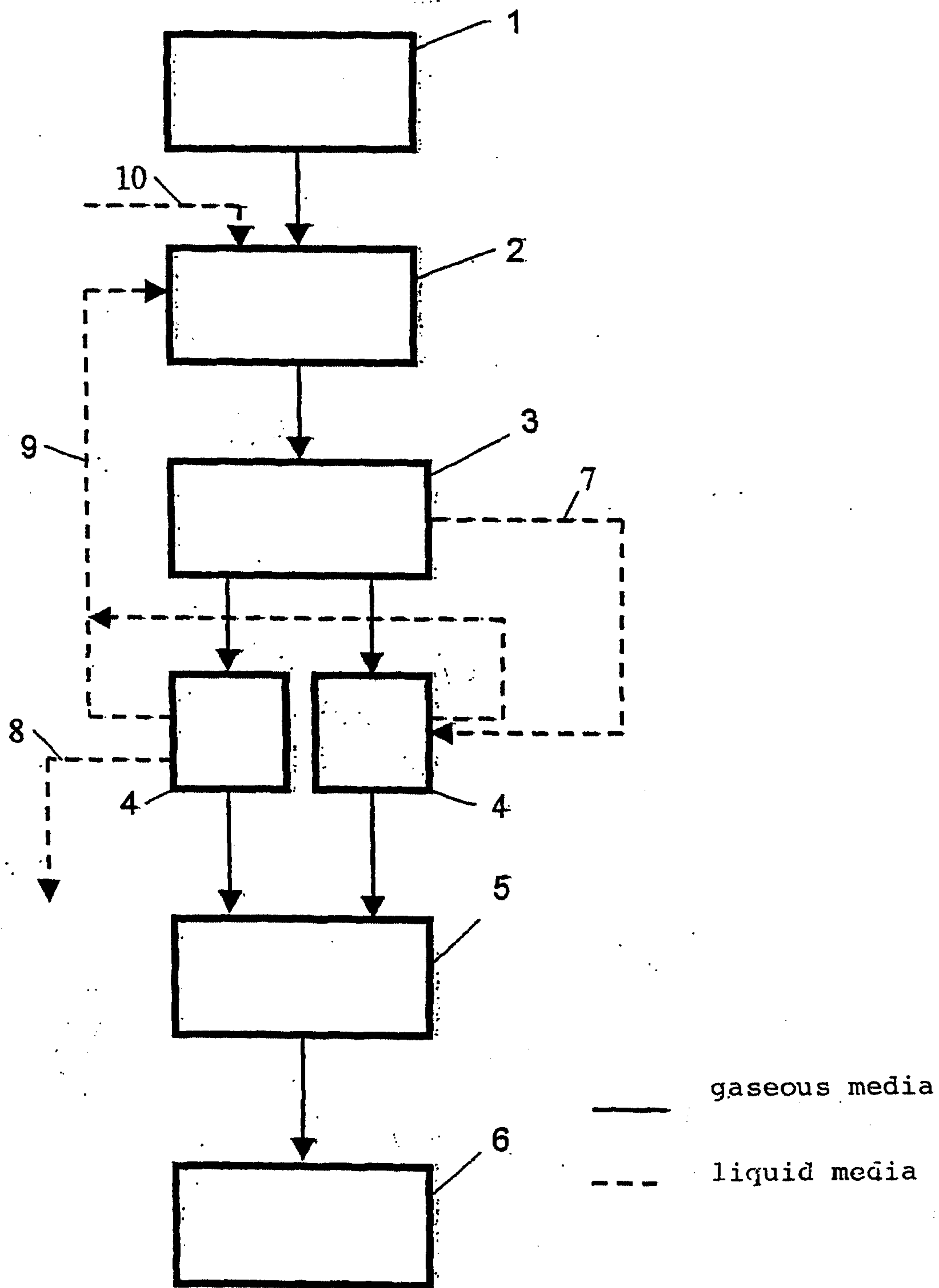
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**Fig. 1**

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**Fig. 2**

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**Fig. 3**