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(54) METHOD FOR PURIFYING TURPENTINE CONDENSATE FROM TRS

(57) The present invention describes a method for cleaning turpentine condensate (1) from total reduced sulphur (TRS) compounds which turpentine condensate is obtained by cooking soft wood pulp in a pulp mill. In the method obtained turpentine condensate (1) is collected and passed to a column (4) supplied with a steam input (5) whereby said steam input contacts said turpentine condensate (1). The TRS compounds is thereby totally or partially removed into a first gas stream (6) which is passed to a condenser (7). The first gas steam (6) is condensed out for obtaining a turpentine condensate (9) which is recycled to said column (4) and a second gas stream (11) is passed to a second condenser (12) from which a condensate (15) is obtained containing impure TRS-containing condensate. At the bottom of column (4) a purified turpentine condensate (17) is obtained and removed.

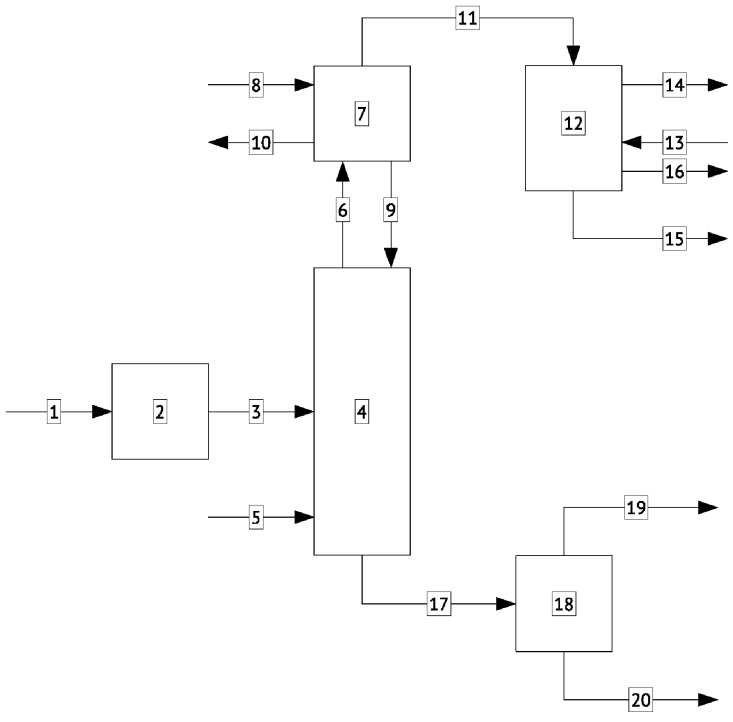

Fig. 1

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Description

Field of the invention

[0001] The present invention relates to a method for purifying turpentine condensate from reduced sulphur compounds, so called TRS (Total Reduced Sulphur).

Background of the invention

[0002] In pulp mills when cooking soft wood, the main portion of the turpentine is normally obtained in the turpentine recovery system of the digester house.

[0003] The amount of turpentine recovered varies widely and is due to a number of more or less complex factors. Among the most important are wood species, the zone and origin of growth of the wood, the storage time of the wood and wood chips, the type of digester process, how the turpentine recovery equipment is designed and its operating conditions, etc.

[0004] Although, most of the turpentine is recovered from the turpentine recovery system of the digester house varying amounts of turpentine reach subsequent processes of the pulp mill, especially the pulp washing sections and the liquor evaporation plant of the pulp mill.

[0005] In the evaporation plant, the extraction liquors of the digester house are thickened from thin black liquor to heavy black liquor, while condensate with different purity grades are extracted.

[0006] The amount of turpentine extracted within the evaporation plant is in most cases substantially lower than the amount of turpentine extracted in the turpentine recovery system of the digester house. Within the evaporation plant the most common location for turpentine extraction is within the methanol recovery system but can be accomplished by other methods within the evaporation plant as well.

[0007] However, in general the turpentine extracted from liquid methanol is considered too contaminated for further sale, mainly because of high sulphur content (TRS). For this reason, this foul turpentine is usually combined with the liquefied methanol for further combustion and destruction. The normal procedure is that recovered liquid methanol, with or without foul turpentine, is used as a support fuel in the pulp mill combustion process for collected concentrate non-condensable gases. By removing the turpentine from the methanol, the heat load with respect to added liquid fuel can be significantly reduced which would be considered an advantage in most cases.

[0008] The inventive method for purifying turpentine from reduced sulphur can either be operated as a batch process or a continuous process depending on specific circumstances. By the inventive method, turpentine condensate streams originating from a soft wood pulp process can be purified from reduced sulphur compounds (TRS).

[0009] Primarily, the inventive method for purification with respect to TRS relates to turpentine condensate streams found from the methanol recovery system of the chemical recovery plant, the evaporation plant, condensates derived from the handling of diluted and concentrated non-condensable gas or similar turpentine condensate streams found within the pulp mill. The pulp mill is intended for processing soft wood pulp and refers to a process for exploit of sulphate pulp, sulphite pulp and processes for the extraction of so-called NSSC, CTMP and TMP pulp.

[0010] Thus, the inventive method is applicable to any turpentine condensate with high sulphur content within the pulp mill.

[0011] However, it is known that even turpentine condensate obtained in the turpentine recovery system of the digester house can occasionally contain high sulphur content. Thus, the inventive method can be applied to turpentine condensates obtained from the turpentine recovery system of the digester house as well if high sulphur content is a problem.

[0012] The inventive method for sulphur purification of turpentine condensates consists of a number of sub-processes where the customer requirement regarding the sulphur content of the purified turpentine determines whether subsequent sub-processes are necessary or not. Thus, the quality requirement for a customer may be that only one of the sub-processes is required to obtain sufficiently pure turpentine, while the quality requirement of another customer requires that the system is designed with all the described sub-processes.

[0013] Thus, due to the fact that the economic value of turpentine has increased in recent years it has become of interest to obtain turpentine even from high sulphur containing turpentine condensate streams and/or other streams containing turpentine.

[0014] Furthermore, by obtaining a larger portion of turpentine from pulp mill condensates the amount of emission of CO_2 to the environment is reduced, since it would otherwise be used as fuel.

[0015] Thus, the object of the invention is to increase the amount of turpentine that can be extracted from TRS containing condensates within a pulp mill producing soft wood pulp. According to the invention, this object is achieved by a method for cleaning turpentine condensates from total reduced sulphur (TRS) compounds which turpentine condensate is obtained by cooking soft wood pulp in a pulp mill, characterized by

- obtaining at least one turpentine condensate,
- collecting said at least one turpentine condensate,
- passing said at least one turpentine condensate to a first column;
- supplying a first steam input to said first column so as to bring said first steam input in contact with said at last one turpentine condensate within said first column in such a way that TRS compounds in the supplied at least one turpentine condensate is totally or partially removed into a first gas stream stripped

from the first column,

- passing said stripped first gas stream to a first condenser where said stripped first gas steam is partially condensed out by supplying a first cooling media to said first condenser thereby obtaining a first resultant turpentine condensate,
- recycling said first resultant turpentine condensate to said first column,
- passing a resultant second gas stream from said first condenser to a second condenser where the resultant second gas stream is totally or partially condensed out by supplying a second cooling media to said second condenser,
- obtaining a first resultant condensate from the second condenser comprising an impure TRS-containing condensate,
- obtaining and removing at a bottom of said first column a first purified turpentine condensate, and
- optionally passing said first purified turpentine condensate to a decanter for gravimetric separation of purified turpentine from a first aqueous phase condensate.

[0016] Other embodiments of the invention will be apparent from the accompanying dependent claims.

Brief description of drawings

[0017] The invention will now be described by way of a non-limiting example with reference to the accompanying drawings, in which

- Fig. 1 is a schematic view of a first embodiment of an apparatus for carrying out the inventive method, and

- Fig. 2 is a schematic view of a second embodiment of an apparatus for carrying out the inventive method.

Description of preferred embodiments

[0018] A turpentine containing condensate, below called turpentine condensate 1, to be treated so as to reduce its TRS content is collected and passed by pumping or hydraulically drainage, for instance, to a tank 2. The tank 2 can be vented to an external gas system or to any of the vessels described below (not shown).

[0019] In a first embodiment of the inventive method, said collected turpentine condensate 1 stored in the tank 2, if appropriate, is supplied through pumping or hydraulic drainage, for instance, via a pipe system 3 to a first column 4. A first steam input 5 is supplied to the first column 4 in which said supplied turpentine condensate 3 and said supplied first steam input 5 are brought into contact with each other in such a way that TRS compounds in the supplied turpentine condensate is totally or partially removed by a first gas stream 6 stripped from said first column. As the first steam input 5, either pure steam or TRS containing steam can be used. The first column 4 can be provided with a stripping section with or without a rectifying section depending on prevailing circumstances.

[0020] Said first gas stream 6 stripped from the first column 4 is passed to a first condenser 7 where said first gas stream 6 is partially condensed out by supplying a first cooling media 8 thereby obtaining a first resultant turpentine condensate 9, the condensing heat being extracted through a first outgoing cooling media 10. The first resultant turpentine condensate 9 is returned to the first column 4 in such a way that a reflux is obtained. The returned first resultant turpentine condensate 9 can be returned to the first column 4 by pumping or by hydraulic drainage, for instance. The returned first resultant turpentine condensate 9 can be returned to the first column 4 at one or several different levels of the first column 4.

[0021] A second resultant gas stream 11 from the first condenser 7 is further passed to a second condenser 12 where the second resultant gas stream 11 is totally or partially condensed out depending on inert gas content. A second cooling media 13 is supplied to the second condenser 12 and the condensing heat being extracted through a second outgoing cooling media 14. A first resultant condensate 15 from the second condenser 12 consists of an impure TRS-containing condensate which is advantageously passed towards the pulp mill handling system for foul condensates. A first resultant inert gas stream 16 is advantageously passed towards the pulp mill system for handling of concentrated non-condensable gas. At the bottom of the first column 4 a first purified turpentine condensate 17 is obtained and removed.

[0022] This first purified turpentine condensate is preferably passed to a decanter 18 for gravimetric separation of purified turpentine 19 from the aqueous phase condensate 20. The first purified turpentine condensate 19 can be passed to the same tank as used for storage of turpentine obtained in the turpentine recovery system of the digester house as mentioned above or to a different tank.

[0023] In a second embodiment of the inventive method, in case the first purified turpentine condensate 17 also contains amounts of heavier TRS compounds such as polymeric sulphur compounds, S8 compounds and similar sulphur compounds, said first purified turpentine condensate 17 is passed without passing a decanter, such as decanter 18, to an oxidizing reactor 30 for total or partial oxidation of residual sulphur compounds. An oxidizing agent 31 is added to the oxidizing reactor 30. The oxidizing agent 31 is preferably selected from the group consisting of peroxides such as hydrogen peroxide, oxygen, air, and the like, and commonly used oxidizing agents in the pulping process of a pulp mill. The oxidizing reactor 30 is designed as a vessel so as to obtain a good contact between the first purified turpentine condensate 17 and the oxidizing agent 31 supplied. The reactor 30 may have means which improves contact be-

tween said treated first purified turpentine condensate and the oxidizing agent supplied.

[0024] A resultant TRS-oxidized turpentine condensate 32 from the reactor 30 is pumped or hydraulically drained, for instance, to a second column 33. A second steam input 34 is supplied to the second column 33 in which supplied TRS-oxidized turpentine condensate 32 and the second steam input 34 are brought into contact with each other in such a way that the components of the turpentine are totally or partially evaporated in the form of a second gas stream 35 stripped from the second column 33. As second steam input 34, pure steam is preferably used. The second column 33 can be designed with a stripping section with or without a rectifying section.

[0025] The second gas stream 35 stripped from the second column 33 is passed to a third condenser 36 where the second gas stream 35 is partially condensed out by supplying a third cooling media 37 thereby obtaining a second resultant turpentine condensate 38 while the condensing heat is extracted by a third cooling media 39. Said second resultant condensate 38 is returned to the second column 33 in such a way that a reflux is obtained. Said second resultant turpentine condensate 38 can be returned to the second column 33 by either pumping or hydraulic drainage, for instance. Said second resultant turpentine condensate 38 can be returned to the second column 33 at one or several different levels of the second column 33.

[0026] A second resultant gas stream 40 from the third condenser 36 is further passed to a fourth condenser 41 where said second resultant gas stream 40 is totally or partially condensed out depending on the inert gas content. The fourth condenser 41 is supplied with a fourth cooling media 42 while the condensing heat is extracted through a fourth outgoing cooling media 43. The resultant inert gas phase 44 is advantageously passed towards the pulp mill system for handling of concentrated non-condensable gas or similar position.

[0027] A second purified turpentine condensate 45 from the fourth condenser 41 is a pure TRS-free turpentine condensate which, by preferably pumping or hydraulic drainage, is passed to a second decanter 46 for gravimetric separation of a second purified turpentine 47 from a second aqueous phase condensate 48. Said second purified turpentine 47 can be led to a handling system of the pulp mill for handling turpentine to be sold, while said second aqueous phase condensate 48 is preferably returned to the pulp mill handling system for foul condensates.

[0028] At the bottom of the second column 33, a foul sulphur-containing condensate 49 is removed. The foul sulphur-containing condensate 49 may contain varying amounts of resulting oxidized sulphur compounds of polymeric sulphur compounds, S8 compounds and other heavy TRS compounds. The foul sulphur-containing condensate 49 can preferably be recycled to any of the black liquor streams of the pulp mill chemical recovery process, the pulp mill handling system for foul condensates or similar suitable liquid partial stream of the pulp mill.

Claims

1. A method for cleaning turpentine condensate (1) from total reduced sulphur (TRS) compounds which turpentine condensate is obtained by cooking soft wood pulp in a pulp mill, **characterized by**

   - obtaining at least one turpentine condensate (1),
   - collecting said at least one turpentine condensate (1),
   - passing said at least one turpentine condensate (1) to a first column (4);
   - supplying a first steam input (5) to said first column (4) so as to bring said first steam input in contact with said at last one turpentine condensate (1) within said first column (4) in such a way that TRS compounds in the supplied at least one turpentine condensate (1) is totally or partially removed into a first gas stream (6) stripped from the first column (4),
   - passing said stripped first gas stream (6) to a first condenser (7) where said stripped first gas steam (6) is partially condensed out by supplying a first cooling media (8) to said first condenser (7) thereby obtaining a first resultant turpentine condensate (9),
   - recycling said first resultant turpentine condensate (9) to said first column (4),
   - passing a resultant second gas stream (11) from said first condenser (7) to a second condenser (12) where the resultant second gas stream (11) is totally or partially condensed out by supplying a second cooling media (13) to said second condenser (12),
   - obtaining a first resultant condensate (15) from the second condenser (12) comprising an impure TRS-containing condensate,
   - obtaining and removing at a bottom of said first column (4) a first purified turpentine condensate (17), and
   - optionally passing said first purified turpentine condensate (17) to a decanter (18) for gravimetric separation of purified turpentine (19) from a first aqueous phase condensate (20).

2. The method according to claim 1, **characterized by** said at least one turpentine condensate (1) comprises a content of total reduced sulphur (TRS) compounds of at least 1 percentage by weight.

3. The method according to claim 1 or 2, **characterized by** said first steam input (5) to said first column (4) being either pure steam or a TRS containing steam.

4. The method according to any one of the preceding claims, **characterized by** providing said first column (4) with a stripping section with or without a rectifying section.

5. The method according to any one of the preceding claims, **characterized by**, in case said first purified turpentine condensate (17) is not passed to said first decanter (18), further comprising:

- passing said first purified turpentine condensate (17) to an oxidizing reactor (30) for total or partial oxidation of residual sulphur compounds in said purified turpentine condensate (17), and
- providing said oxidizing reactor (30) with at least one oxidizing agent (31), said oxidizing agent being selected from the group consisting of peroxides such as hydrogen peroxide, oxygen, air, or the like commonly used oxidizing agents within the pulp mill, for obtaining a resultant TRS-oxidized turpentine condensate (32),
- passing said resultant TRS-oxidized turpentine condensate (32) from said oxidizing reactor (30) to a second column (33),
- supplying a second steam input (34) to said second column (33) so as to bring said TRS-oxidized turpentine condensate (32) and said second steam input (34) in contact with each other so that turpentine components of said resultant TRS-oxidized turpentine condensate (32) are totally or partially evaporated into a second gas stream (35) stripped from said second column (33),
- passing said stripped second gas stream (35) to a third condenser (36) where said stripped second gas stream (35) is partially condensed out by supplying a third cooling media (37) to said third condenser (36),
- recycling a second resultant turpentine condensate (38) to said second column (33),
- passing a second resultant gas stream (40) from said third condenser (36) to a fourth condenser (41) where said second resultant gas stream (40) is totally or partially condensed out,
- obtaining a second resultant condensate (45) from the fourth condenser (41) which is a pure TRS-free turpentine condensate, and
passing said second resultant condensate (45) to a second decanter (46) for gravimetric separation of a second purified turpentine (47) from a second aqueous phase condensate (48).

6. The method according to claim 5, **characterized by** returning said second aqueous phase condensate (48) to a pulp mill handling system for foul condensates.

7. The method according to claim 5 or 6, **characterized by**

- removing a dirty sulphur-containing condensate (49) at the bottom of said second column (33), and
- recycling said foul sulphur-containing condensate (49) to any black liquor streams of a chemical recovery unit, the pulp mill handling system for foul condensates or similar suitable liquid partial stream of the pulp mill.

8. The method according to any one of the preceding claims, **characterized by** cooking said soft wood pulp by a process so as to obtain a pulp selected from the group consisting of a sulphate pulp, sulphite pulp, NSSC pulp, CTMP and TMP.

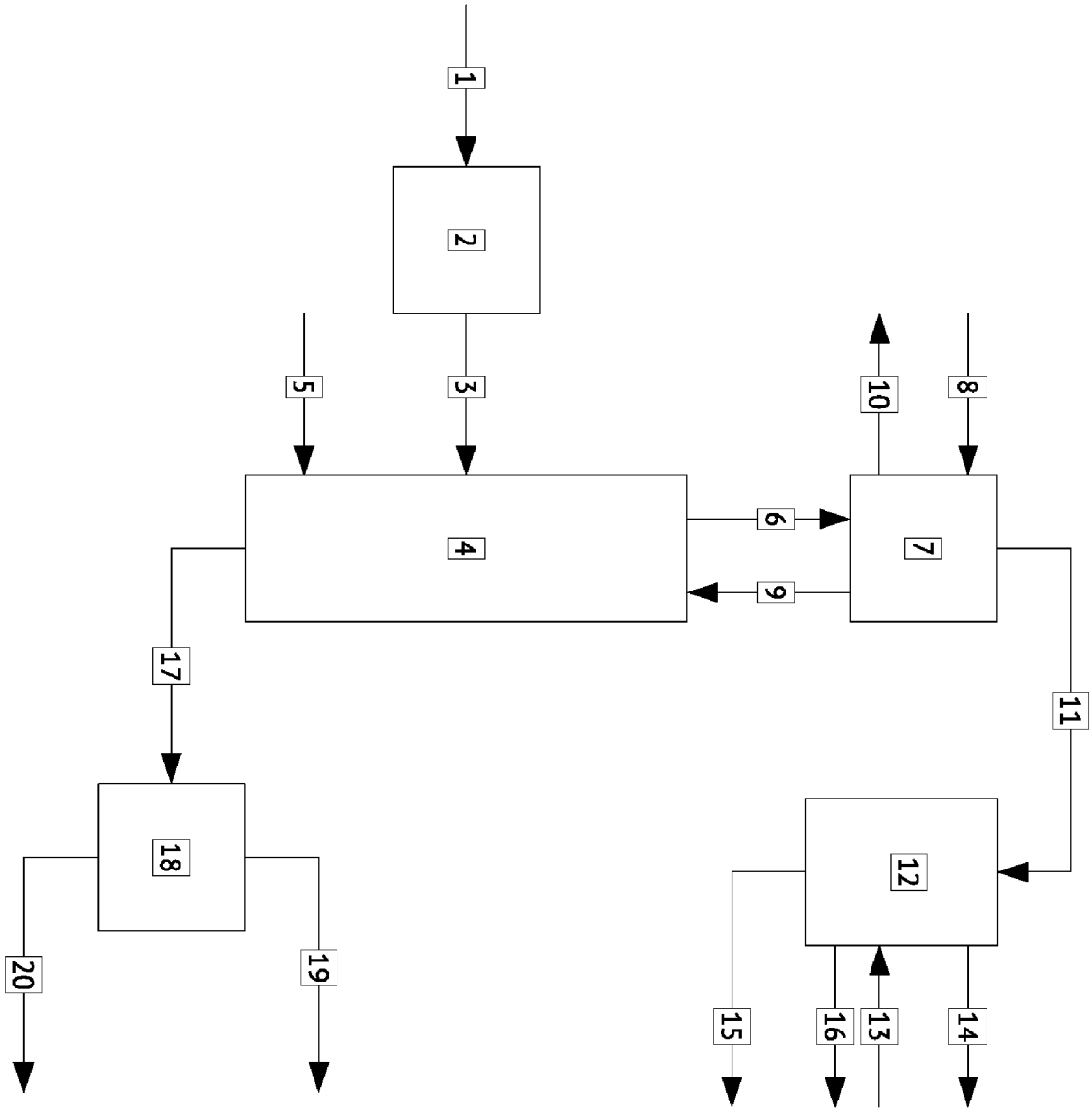

Fig. 1

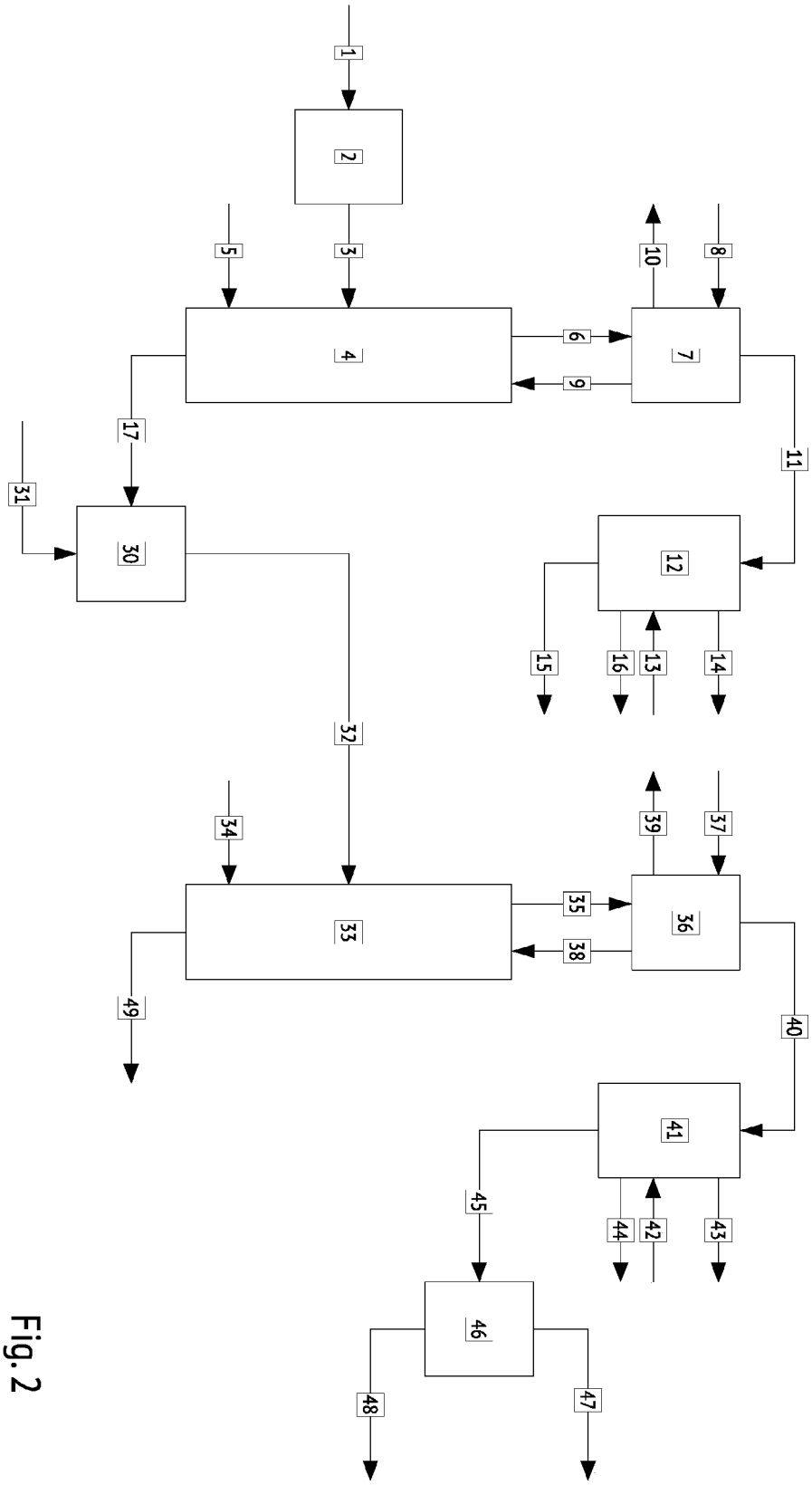
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Fig. 2

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EUROPEAN SEARCH REPORT

Application Number
EP 19 21 1806

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	7 February 2020	Karlsson, Lennart

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