PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Method of Recovering Solvents from Waste Gases

We, METALLGESELLSCHAFT AKTIENGESELLSCHAFT, of 45, Bockenheimer Anlage, Frankfurt-on-the-Main, Germany, a Corporation organised under the Laws of Germany, 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 a method of recovering solvents from waste gases obtained in extraction processes.

In the extraction of valuable substances, such as phenols, fatty acids, alcohols, organic bases or the like, from waste liquors or aqueous solutions, by means of organic solvents, e.g., benzene or benzol, organic esters, ethers, ketones or the like, which are insoluble or difficultly soluble in the said waste liquors or aqueous solutions a part of the solvent remains in the liquor or aqueous solution after the separation from said waste liquors or aqueous solutions of the solvent charged with the extracted substances. From the thus treated liquors the residual solvents still contained therein are recovered, for example by distillation or by pressure reduction if the extraction process has been carried out under pressure or by application of a vacuum. In recovering the solvent from the treated liquors, gases which have been dissolved in said liquors are simultaneously set free and larger or smaller amounts of the solvent pass into these gases.

Various processes have been used for recovering the solvent. For instance in the extraction of waste water of coking plants with benzol, the liberated gas was added to the coke over gases before subjecting them to the usual benzol recovery treatment Where aqueous liquids resulting from dry distillation of solid fuel were extracted with organic oxygen-containing compounds such as esters or ketones, for the purpose of recovering phenols or the like therefrom, the waste gases produced by the recovery of the solvent from the extracted liquids were treated with the liquids supplied to the extraction apparatus.

According to the present invention the bulk of the solvent is removed in vapour form from the aqueous liquid by distillation, pressure reduction, vacuum or the like, the solvent vapours thus formd are separately condensed and the residual gases formed during the removal of the solvent from the aqueous liquid and remaining after the condensation of the solvent vapours are freed from residual solvent still contained therein by washing said gases with the whole or a part of the extracted substance freed from solvent.

It has been found that the extracted substances are excellent absorbing agents for the recovery from the waste gases of the vapours of the extracting agents used. Such extracted substances are capable of absorbing from the gases up to 50% and more of their weight of solvent. Accordingly only relatively small quantities of extracted substances are required for recovering the solvents passing over into the waste gases, whereas it was hitherto necessary to scrub the waste gases in a washing tower with the entire quantity of the liquid to be extracted. The mixture of solvent and extracted substances obtained by the process of the present invention can be processed together with the extracted substances, without causing a substantial additional load in the extract distillation plant in which the decomposition of the extract into solvent and extracted substances is carried out.

For instance, if phenols or the like are extracted by means of organic esters such as butyl acetate, from carbonization liquors, coking liquors or the like, the plant illustrated diagrammatically and by way of example in the accompanying drawing can be employed with good results for recovering the solvent from the waste gases produced in the plant.

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Referring to said drawing, 1 denotes a cooler in which the hot phenol-containing water is cooled down to about 30 to 35° C. The cooled water then passes through the pipe 2 to a three-stage counter-current extraction apparatus 3. The extracted water containing residual solvent flows from the apparatus 3 through a pipe 21 into an intermediate tank 4 and is forced by a pump 22 through duct 25, heat exchanger 5 and preheater 6 into a distilling column 11, into the still 10 of which live steam is introduced for heating. From the still 10 the hot water freed from solvent flows through duct 24 into the heat exchanger 5, where the heat of the water is transferred to the extracted water fed through duct 25, the water being discharged in a cooled condition through the duct 23. The gases and vapours escaping from the column 11 flow through a pipe 26 into a condenser 9, where solvents and steam are condensed from the gases. 27 is the cooling water feed duct and 28 the cooling water discharge duct of the condenser 9. The condensate from the condenser 9 is now passed through the apparatus 8 in which liquid and gases are separated from each other in known manner and from thence to a separator 7, in which the water is separated from the solvent. The solvent is passed through the pipe 12 into the reservoir 13, while the water containing residual solvent is passed through pipe 29 to the extracted water in the intermediate tank 4. The gases which have been separated from the condensate in the apparatus 8 now flow through the pipe 30 into the washing tower 31.

The solvent charged with the extracted substances is passed through the pipe 32 to a distilling column 15 of a known construction. The solvent vapours distilled off in the column are passed through the pipe 17 into the condenser 16 where they are liquified, the condensate being returned through pipe 18 into the reservoir 13. From the still 14 of the column 15 the phenols freed from the solvent are passed through the pipe 33 and the cooler 19 to the carbolic acid or phenol tank 20, for further treatment or use in known manner. A part of the phenols is fed, through the duct 34, to the washing tower 31, as washing agent. In this washing tower the phenols absorb the solvent from the waste gases which then leave the plant. Said phenol-solvent mixture is then conveyed by the pump 36 through the duct 37¹ into the duct 32 through which they are supplied together with the extract-containing solvent coming from the extraction plant, to the distilling column 15. From the reservoir 13 the recovered solvent is recycled through the duct 37 into the extraction plant.

For instance, waste liquors occurring in the carbonization of Middle German lignite and containing about 15 grammes of phenol substances per litre, are treated with 100 litres of butyl acetate per cubic metre of waste liquor in the three-stage extraction plant 3. The solvent which has absorbed about 15 kgs. of phenol per 100 litres is separated in the distilling column 15 into solvent and raw phenol. 10% of the raw phenols obtained, i.e., 1.5 kgs. are used for washing the waste gases in the tower 31. The quantity of the waste gases which consist of carbon dioxide and hydrogen sulphide amounts to about 4 cubic metres at NTP per 1 cu. metre of waste liquor. About 100 grs. of butyl acetate are contained in 1 cubic metre of waste gas at NTP. The butyl acetate is washed out of the gas in the washing tower 31 down to 0.04 grammes at NTP (i.e. 99.96%). The washing agent flowing off from the washing tower consists of about 80% by weight of raw phenol and 20% of butyl acetate. This mixture is worked up together with the solvent in the distillation column 15.

The recovery of the vapours of the extracting agent contained in the waste gases can be carried out in a similar manner where other extracting agents are used, and it is also possible to treat the waste gases of other extraction processes in accordance with the invention, the waste gases resulting in the recovery of the solvent also being washed with the extracted substance. The latter may be processed together with the charged solvents in a similar manner.

What we claim is:—

1. A method of recovering solvent which remains behind in a liquor after said liquor has been extracted with said solvent to recover valuable substances, e.g. as in the recovery of phenols from waste liquors by extraction with butyl acetate, which is characterised in that the extracted liquor is distilled to remove the residual solvent, whereupon said residual solvent is condensed and separated from gases formed in the distillation and said gases are freed from residual solvent contained therein by washing with the valuable substances which have been recovered and separated from said solvent.

2. Method as claimed in Claim 1, in which the mixture of solvent and valuable substances produced after washing out the residual gases together with the solution of valuable substances obtained by the extraction from the extracted liquor containing solvent are worked up

in the solvent for separation into solvents and valuable substances.

3. Method as claimed in Claim 2, in which the working up is effected in a column still.

4. Method as claimed in Claim 3, in which the mixture of solvent and valuable substances obtained after washing the gases is led into a part of the column still in which the downwardly flowing solution obtained by the extraction of the solutions of valuable substances obtained in the solvent has approximately the same concentration as the mixture to be introduced.

5. Method as claimed in any of Claims 1 to 4, applied to the treatment of phenol-containing aqueous solutions which have been produced in the distillation or hydrogenation of fuels.

6. Method of recovering solvents from waste gases, substantially as described with reference to the accompanying drawing.

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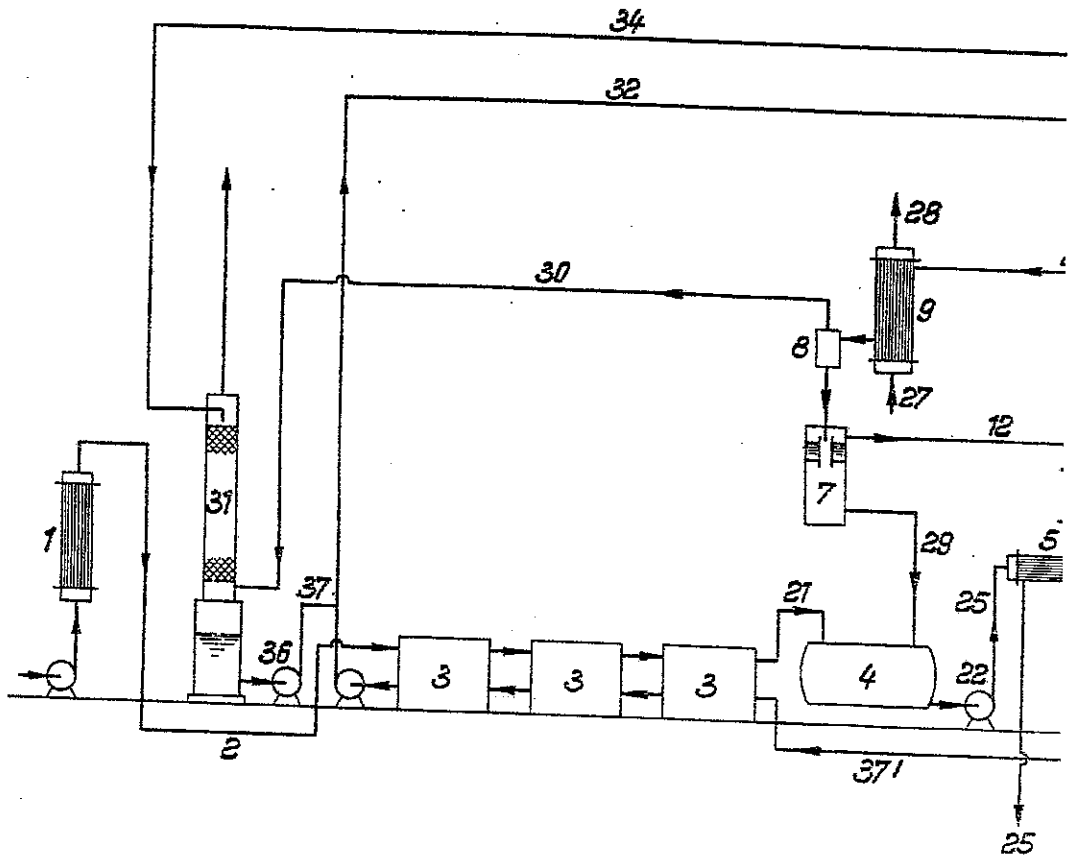
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688,228 COMPLETE SPECIFICATION

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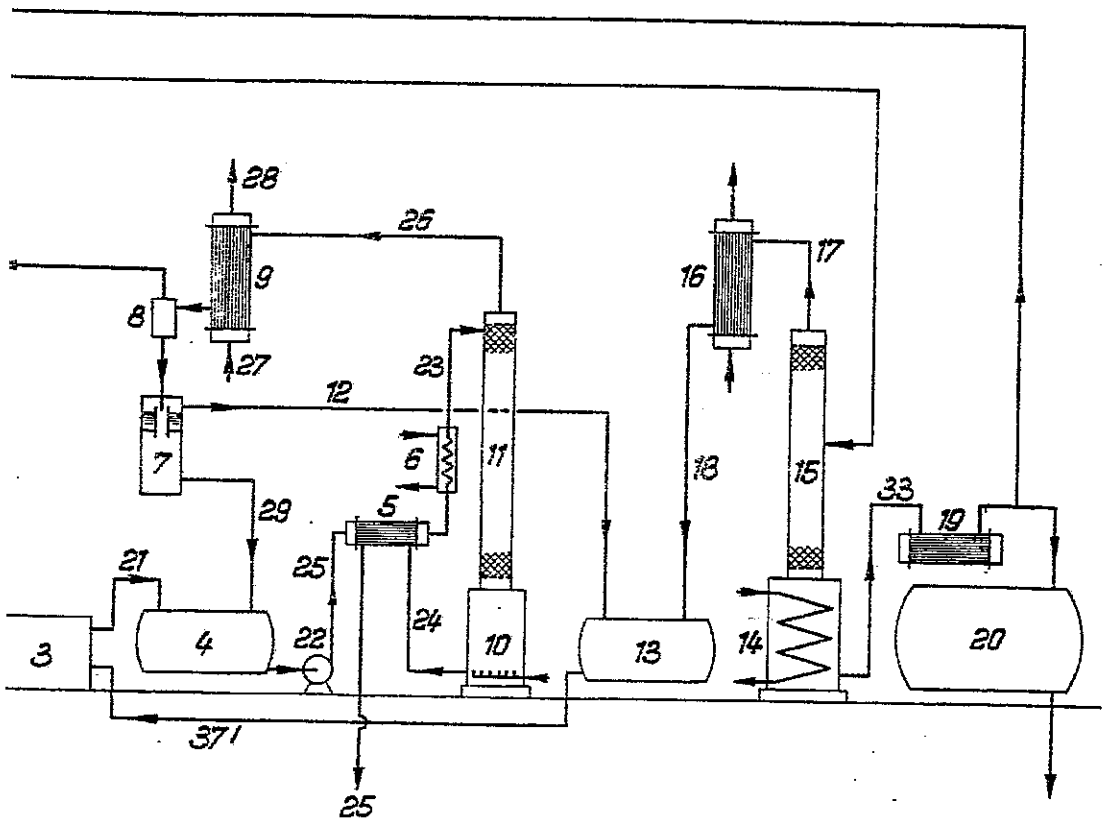
This drawing is a reproduction of the Original on a reduced scale.



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