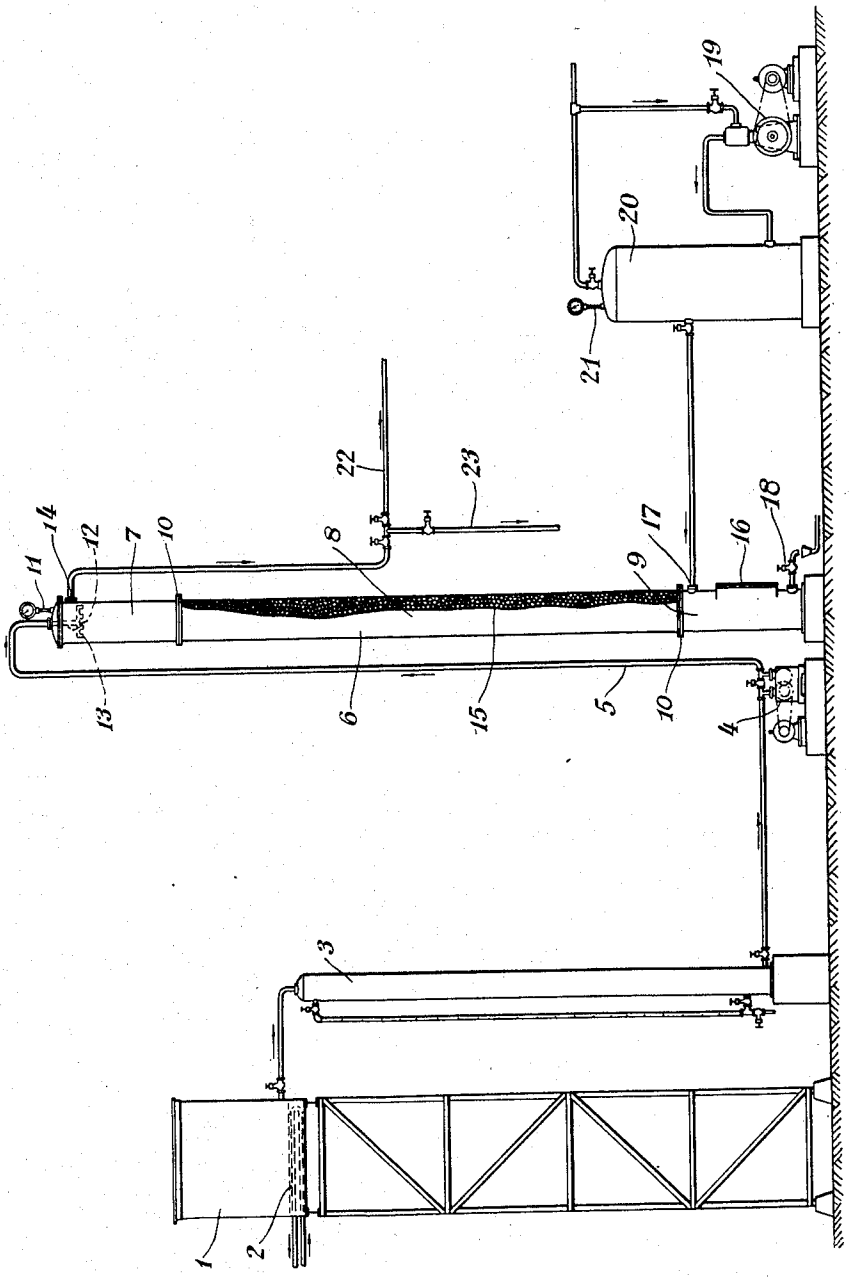
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RECOVERY OF TAR ACIDS FROM COAL OILS OR TARS

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RECOVERY OF TAR ACIDS FROM COAL OILS OR TARS

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1 Claim. (Cl. 260—627)

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The invention relates to a process for the recovery of tar acids from coal oils or tars which may be derived from high or low temperature carbonisation or distillation processes in which the oil or tar is first washed with caustic soda to take the tar acids into solution and the tar acids then extracted from the solution or cresylate by springing or acidification with carbonic acid gas.

Among the disadvantages of such processes are the inefficient use of the carbon dioxide gas, of which only about 60% takes part in the reaction; at the end of the reaction an average of about 12% of carbonate or unreacted cresylate remains in the separated tar acids and about 4% of tar acids or unreacted cresylate in the carbonate. Thus for final springing or acidification a relatively large volume of mineral acid is required with a correspondingly large loss of sodium carbonate.

According to the invention, the extraction of the tar acids from the solution or cresylate is carried out at pressures of 30–35 lbs. per sq. in. The solution or cresylate produced in the washing of the coal oils or tars with caustic soda may be passed in countercurrent with gaseous carbon dioxide of a concentration of about 15–20% through towers packed with Raschig rings.

The process of the invention was carried out in apparatus as diagrammatically illustrated by way of example in the accompanying drawing.

From a storage vessel 1 heated by means of steam pipes 2 the solution or cresylate from the washing of the coal oils or tars with caustic soda was passed through a calibrated vessel 3 for measuring the rate of flow, and then through a proportion-meter pump 4 by way of a pipe 5 to the top of a column 6 whence it passed downwardly in countercurrent with carbonic acid gas passing upwardly through the column. The column 6 which was of 3'' diameter, was made up in three sections 7, 8, 9, joined together with pressure-tight flanges 10. The top section 7, 1' in length, was fitted with a pressure gauge 11 and provided with an inlet 12 for the feed from the storage vessel 1, provided with a distributor 13 to ensure an even distribution of the cresylate over the packing, and an outlet 14 for the gases passing upwardly through the column 6. The middle section 8, of the column 6, five feet in length, was packed with 3/8'' Raschig rings 15. The lowermost section 9, 1' in length, was fitted with a

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sight glass 16 and provided with an inlet 17 for the carbonic acid gas and a run-off valve 18 for the acidified or sprung cresylate.

The carbonic acid gas supplied to the column 6 contained 20% CO_2 and was passed through a compressor 19 to a storage tank 20 provided with a pressure gauge 21 at a pressure slightly above the pressure to be maintained in the column 6.

The outlet gases from the top of the column 6 flow either through a CO_2 recorder by way of pipe line 22, or through a gas flow meter by way of pipe line 23.

In operation the column 6 was first adjusted to the pressure required by means of a by-pass circuit on the storage tank of the carbonic acid gas, whereby the pressure in the storage tank was maintained at about 32 lbs./sq. in. and in the column at about 30 lbs./sq. in.

The flow of the cresylate to the top of the column 6 was controlled by the proportion-meter pump 4.

The column was operated for between one to two hours to ensure stability of operating conditions. The chemical reaction being exothermic, resulted in a heating of parts of the column. After stabilisation of the operating conditions, the volume of flow of carbonic acid gas was slowly reduced until the exit gases contained from 1 to 2% CO_2. After the lapse of a period for conditions to become settled, samples of the tar acids and sodium carbonate were removed from the bottom of the column.

Tests on the tar acids and carbonate solution produced were carried out as follows:

To 50 cc. of a 50% "brown oil of vitriol" solution were added 50 cc. of the tar acids and intimately admixed in a stoppered vessel. After separation into two layers, the increase in volume of the mineral acid layer multiplied by two, will give an indication as to the percentage carbonate in the tar acids.

50 cc. of carbonate solution were acidified in a standard tar acid flask, with "brown oil of vitriol" to which was added 5 cc. of tar acids to assist in the separation of the tar acids from the acidified carbonate. The volume of tar acids in cc. less the five cc. of tar acids added, and the result multiplied by two, will give an indication of the percentage tar acids in the carbonate.

The results obtained during three runs are tabulated below:

Exp.	Press in Column, lbs./sq. in.	Temp. of Inlet Cresyl., °C.	Rate of Feed, cc./min.	Per cent CO_2 in inlet	Per cent CO_2 in gas outlet	Gas throughput, c. ft./hr.	T. A. in Carb., Per cent	Carb. in T. A., Per cent
1	Atmos.	24	100	20	8	27	4.8	16
2	30	25	100	20	2	31	2	2
3	35	30	100	20	1.5	30	2	3

The advantages to be secured from the process of the invention are clear from a comparison of the results of the first experiment carried out at atmospheric pressure and the second and third experiments carried out according to the process of the invention at pressures in the operating column respectively of 30 lbs./sq. in and 35 lbs./sq. in.

We claim:

A continuous process for the recovery of tar acids from coal tars, which comprises the following sequential steps: reacting coal tar with an aqueous solution of sodium hydroxide to form an aqueous solution of the sodium salt of the tar acids; separating said aqueous solution from the remainder of the tar; introducing said aqueous solution into one end of an extraction zone; introducing a gas mixture containing not more than 20% of carbon dioxide into the other end of said zone; passing said mixture and said solution through said zone in countercurrent flow,

whereby free tar acids are liberated, while maintaining the pressure in said zone within the range of 30–35 lbs. per sq. in.; withdrawing from said other end of said zone the resulting mixture of free acids and aqueous solution of sodium carbonate; and separating said free acids from said carbonate solution.

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