

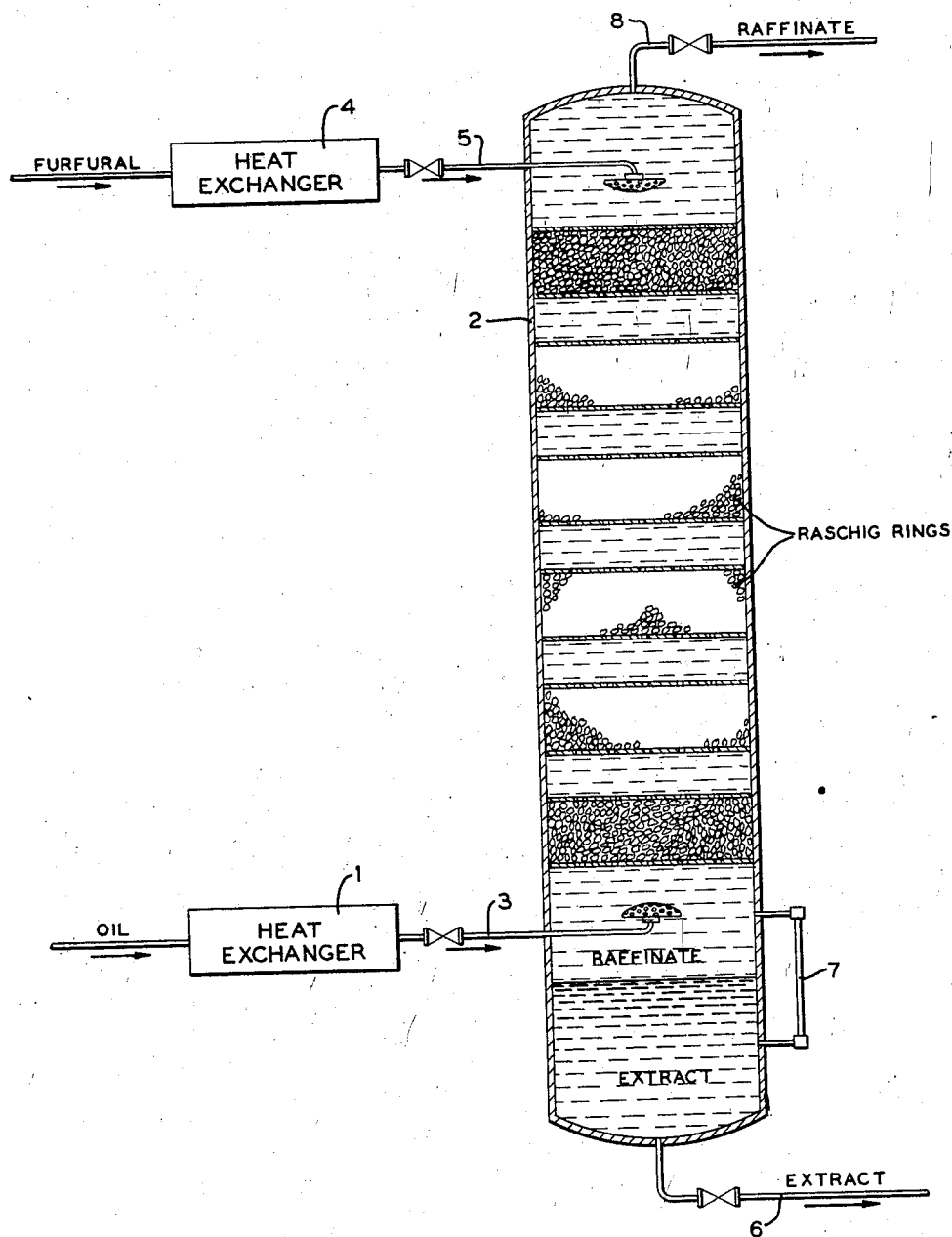
Sept. 10, 1940.

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2,214,282

SOLVENT REFINING OF MINERAL OIL

Original Filed June 14, 1934



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2,214,282

SOLVENT REFINING OF MINERAL OIL

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Continuation of application Serial No. 730,575,
June 14, 1934. This application May 8, 1937,
Serial No. 141,464

8 Claims. (Cl. 196—13)

This invention relates to a method of refining hydrocarbon oil, particularly mineral oil, with selective solvents.

This application is a continuation of our co-pending application, Serial No. 730,575, filed June 14, 1934.

The invention has to do with the extraction of mineral oil containing naphthenic and paraffinic constituents with a selective solvent of the character of furfural, whereby the oil is separated into fractions respectively rich in paraffinic and naphthenic constituents.

The invention contemplates a method of extraction wherein the oil is brought into contact with furfural in the form of a relatively thin film disposed over exposed contact surfaces and over which the oil is caused to flow in relatively divided form. The invention is adaptable to a counter-current type of operation, such as may be carried out in a vertical treating zone, containing a suitable packing material adapted to expose a relatively large area of surface for contact with the oil and solvent.

We have found that, in the presence of mineral oil, furfural possesses a preferential wetting action for the exposed surfaces of packing material of the character of Raschig rings, tile, pebbles, and the like, even though the packing material is submerged or surrounded with mineral oil. Thus, it has been found that when furfural is introduced to the top of a packed tower maintained full of oil, the furfural, which is of relatively heavier specific gravity, in descending through the body of oil and packing material, tends to adhere to the exposed surfaces of the packing material in the form of a relatively thin film.

Where the packing material is maintained submerged in a body of furfural, rather than a body of oil, the foregoing film effect is not realized. The oil rises in the form of particles or droplets through a relatively large body of solvents so that a relatively large amount of solvent is required to obtain a given degree of extraction.

We have discovered that when advantage is taken of this preferential wetting action of furfural in order to realize contact between the oil and the solvent disposed as a film, more effective separation of the oil into naphthenic and paraffinic fractions results, and that the solvent is also used more efficiently.

In order to more clearly describe the invention, reference may be made to the accompanying drawing forming a part of the specification.

The oil to be extracted is conducted from a source not shown, through a heat exchanger 1,

wherein it is brought to the desired temperature and introduced to the lower portion of the extraction tower 2, through a pipe 3. The oil is introduced at a rate sufficient to maintain the tower full of oil.

Furfural is conducted from a source not shown through a heat exchanger 4, wherein it is brought to the desired temperature, and thereafter introduced to the upper portion of the tower 2 through a pipe 5.

The tower 2 is packed with Raschig rings or other suitable packing material adapted to expose a relatively large area of contact surface.

The furfural is sprayed over the top of the upper layer of packing material in a proportion suitable to maintain the desired film of solvent moving over the exposed surfaces of the packing material. The oil, as it rises through the packing material, is brought into very intimate contact with the film of solvent disposed over the packing material. By filling the extraction tower full of oil and operating as above described, the solvent thus moves through the oil in a relatively dispersed phase with respect to the oil.

The furfural dissolves the naphthenic bodies and accumulates in the lower portion of the tower where it separates from the main body of the oil as an extract layer. The surplus extract accumulated in the bottom of the tower is drawn off through a pipe 6. The rate at which it is drawn off may be controlled by means of the sight glass 7 so as to maintain a substantially constant level of the extract in the bottom of the tower. Thus, the interface between extract and raffinate phases is maintained at a point low in the tower rather than near the top of the tower so that there is maintained within the tower a body of oil in the continuous phase while the solvent is in the dispersed phase. By maintaining the level between extract and raffinate phases at a point low in the tower, the efficiency of the tower is very substantially improved, particularly when operating the tower at or near its capacity.

The raffinate or paraffinic oil relatively insoluble in the furfural rises to the top of the tower and overflows therefrom through a pipe 8.

In actual operation, the extraction may be carried out at elevated temperatures of around 180° to 225° F. Furfural exerts its optimum selective action upon mineral oil at around these temperatures, and which is of particular advantage since it readily permits extraction of viscous lubricating oils in a single treating zone of the type above described. Viscous oils, particularly wax-bearing oils, are in a sufficiently fluid condition at

these temperatures to permit intimate contact between oil and solvent in this type of apparatus.

While the desired temperatures may be maintained by the application of external heating to the extraction tower, nevertheless this can be more advantageously effected by preliminary heating of either the oil or the solvent, or both, prior to introduction to the tower as already described.

In some instances it may be desirable to maintain a temperature differential throughout the treating zone, namely, to maintain the upper portion of the tower at a relatively higher temperature than the lower portion. In such case, the furfural may be introduced to the top of the tower at a temperature of around 200° or 300° F., while the oil is introduced to the bottom of the tower at a temperature of around 100° to 150° F.

Different temperature conditions may be maintained, of course, depending upon the nature of the oil undergoing treatment. For example, it may be desirable, in some instances, as when treating relatively non-viscous oils, to employ temperatures of 100° F. or below.

Furthermore, it may be of advantage, particularly in the case of relatively viscous residual cylinder stocks, to effect the extraction in the presence of a suitable diluent or modifying solvent. It is contemplated that a suitable solvent for this purpose may comprise a light petroleum fraction, such as naphtha or a normally gaseous fraction of the character of propane, butane, etc. Aromatic hydrocarbon solvents, such as benzol and its homologs may be used, if desired. Relatively low-boiling alcohols, ethers, and the like, may also be employed.

The presence of this diluent or modifying material reduces the viscosity of the oil and permits more effective contact between oil and extraction solvent, and also facilitates the separation into the raffinate and extract layers within the treating zone.

The diluent or modifying solvent may be mixed with the oil in suitable proportion prior to introduction to the tower. Where relatively low-boiling diluent solvents, such as propane, are used, sufficient pressure is maintained within the treating zone to keep the diluent in a liquid state.

As an example of the operation of our invention a lubricating oil distillate derived from Mid-Continent crude and having the tests which will be indicated later was subjected to countercurrent extraction with furfural in a packed tower.

In commencing the run, the tower was first filled with oil. With the tower full of oil, oil and furfural were then continuously co-charged to the tower, the oil being charged near the bottom thereof while the furfural, at a higher temperature, was introduced to the upper portion thereof. The top of the tower was maintained at a temperature of 215° F., while the temperature at the bottom was 154° F. In this way, a temperature gradient of 61° F. was maintained throughout the tower.

The ratio of solvent to oil continuously co-charged was 157 parts of solvent to 100 parts of oil.

Throughout the period during which oil and solvent were continuously co-charged to the tower, the extract level was maintained near the bottom of the tower by visual inspection of the gauge glass 7, shown in the drawing. This was accomplished by continuously withdrawing the extract and the raffinate phase from the bottom

and top of the tower respectively at a rate such that their combined volume was substantially equivalent to the combined volume of oil and solvent being continuously co-charged to the tower. In this way, the tower can be maintained relatively full of oil irrespective of the ratio of solvent to oil co-charged.

After stripping the solvent from the extract and raffinate phase mixtures withdrawn from the tower, the resulting extract and raffinate oil was found to have the following laboratory tests as compared with the charge oil:

	Charge	Extract	Raffinate
Gravity.....	21.8	4.9	24.7
S. U. vis. @ 210° F.....	79	362-359	72-73
Percent carbon residue.....	1.19	9.22	0.64
Viscosity index.....	59	-----	75

The method of our invention, wherein the oil is maintained in the continuous phase and the solvent in the dispersed phase within a countercurrent extraction tower by maintaining the level between the extract and raffinate phases at a point relatively low in the tower, is also applicable to extraction operations wherein other selective solvents are employed, as, for example, nitrobenzene, dichlorethyl ether, phenol, benzaldehyde, etc.

It is also desirable when employing such solvents to maintain within the extraction tower a temperature gradient as above described, although the actual terminal temperatures and the extent of the temperature differential will depend upon the nature of the selective solvent used as well as upon the oil and the type of extraction desired. The maintaining of a wide temperature differential greatly improves the yield of high viscosity index raffinate.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. In the countercurrent extraction of hydrocarbon oil with a selective solvent to effect separation between naphthenic and paraffinic constituents of the oil, the method comprising introducing the oil to a packed vertical extraction tower, introducing the solvent thereto at a point remote from the point of introduction of the oil and at a temperature at least about 50° F. higher than that of the entering oil, maintaining a temperature gradient of 50 to 200° F. throughout the tower, the solvent inlet portion of the tower being at the higher temperature, maintaining within the tower a body of oil in relatively continuous phase with respect to the solvent, moving the solvent countercurrently through said oil while in a relatively dispersed phase with respect to the oil, forming within the tower an extract phase containing naphthenic constituents dissolved in the bulk of the solvent and a raffinate phase comprising paraffinic constituents of the oil mixed with some of the solvent, maintaining the extract phase level relatively near the extract outlet from the tower irrespective of the ratio of solvent to oil continuously co-charged to the tower, and separately withdrawing said extract and raffinate phases from the tower.

2. The method of countercurrently extracting mineral lubricating oil containing low and high

viscosity index constituents comprising introducing the oil to the lower portion of an extraction tower, introducing a solvent having selective action as between low and high viscosity index constituents of the oil to the upper portion of the tower at a temperature at least about 50° F. higher than the temperature of the entering oil maintaining a temperature gradient of 50 to 200° F. throughout the tower, the solvent inlet portion of the tower being at the higher temperature, forming extract and raffinate phases within the tower, maintaining within said tower a volume of oil relatively large with respect to the volume of the solvent, maintaining the extract phase level relatively near the bottom of said tower irrespective of the ratio of solvent to oil continuously co-charged to the tower, removing an extract phase containing low viscosity index constituents from the bottom of said tower, and removing a raffinate phase containing high viscosity index constituents from the top of the tower.

3. The method of extracting mineral lubricating oil containing low and high viscosity index constituents comprising introducing the oil to the lower portion of an extraction tower, introducing an extraction solvent having selective action as between low and high viscosity index constituents of the oil to the upper portion of the tower at a temperature relatively higher than the temperature of the entering oil, maintaining within said tower a volume of oil relatively large with respect to the volume of the solvent irrespective of the ratio of oil to solvent continuously co-charged to the tower, moving the solvent countercurrently through the oil in a relatively dispersed phase with respect to the oil, maintaining a temperature differential of from 50° to 200° F. through the said tower between the points of solvent and oil introduction, removing an extract phase containing low viscosity index constituents from the bottom of said tower, and removing a raffinate phase containing high viscosity index constituents from the top of the tower.

4. The method of extracting mineral lubricating oil containing low and high viscosity index constituents comprising diluting the oil, introducing the diluted oil to the lower portion of an extraction tower, introducing an extraction solvent having selective action as between low and high viscosity index constituents of the oil to the upper portion of the tower at a temperature relatively higher than the temperature of the entering oil, maintaining within said tower a volume of oil relatively large with respect to the volume of solvent irrespective of the ratio of oil to solvent continuously co-charged to the tower, moving the solvent countercurrently through the oil in a relatively dispersed phase with respect to the oil, maintaining a temperature differential of from 50° to 200° F. throughout said tower between the points of solvent and oil introduction, removing an extract phase containing low viscosity index constituents from the bottom of said tower, and removing a raffinate phase containing high viscosity index constituents from the top of the tower.

5. The method of countercurrently extracting mineral lubricating oil containing low and high viscosity index constituents comprising introducing the oil to the lower portion of an extraction tower, introducing a solvent having selective action as between low and high viscosity index constituents of the oil to the upper portion of the tower at a temperature relatively higher than the temperature of the entering oil, forming ex-

tract and raffinate phases within the tower, maintaining the extract phase level relatively near the bottom of said tower irrespective of the ratio of solvent to oil continuously co-charged to the tower, maintaining a temperature differential of from 50° to 200° F. throughout said tower between the points of solvent and oil introduction, removing an extract phase containing low viscosity index constituents from the bottom of said tower, and removing a raffinate phase containing high viscosity index constituents from the top of the tower.

6. The method of extracting mineral lubricating oil containing low and high viscosity index constituents comprising introducing the oil to the lower portion of an extraction tower, introducing a solvent comprising furfural to the upper portion of the tower at a temperature relatively higher than the temperature of the entering oil, maintaining within said tower a volume of oil relatively large with respect to the volume of solvent irrespective of the ratio of oil to solvent continuously co-charged to the tower, moving the solvent countercurrently through the oil in a relatively dispersed phase with respect to the oil, maintaining a temperature differential of from 50° to 200° F. throughout said tower between the points of furfural and oil introduction, removing an extract phase containing low viscosity index constituents from the bottom of said tower, and removing a raffinate phase containing high viscosity index constituents from the top of the tower.

7. The method of extracting mineral lubricating oil containing low and high viscosity index constituents comprising diluting the oil, introducing the diluted oil to the lower portion of an extraction tower, introducing furfural to the upper portion of the tower at a temperature relatively higher than the temperature of the entering oil, maintaining within said tower a volume of oil relatively large with respect to the volume of solvent irrespective of the ratio of oil to solvent continuously co-charged to the tower, moving the solvent countercurrently through the oil in a relatively dispersed phase with respect to the oil, maintaining a temperature differential of from 50° to 200° F. throughout said tower between the points of furfural and oil introduction, removing an extract phase containing low viscosity index constituents from the bottom of said tower, and removing a raffinate phase containing high viscosity index constituents from the top of the tower.

8. The method of countercurrently extracting mineral lubricating oil containing low and high viscosity index constituents comprising introducing the oil to the lower portion of an extraction tower, introducing a solvent comprising furfural to the upper portion of the tower at a temperature relatively higher than the temperature of the entering oil, forming extract and raffinate phases within the tower, maintaining the extract phase level relatively near the bottom of said tower irrespective of the ratio of solvent to oil continuously co-charged to the tower, maintaining a temperature differential of from 50° to 200° F. throughout said tower between the points of furfural and oil introduction, removing an extract phase containing low viscosity index constituents from the bottom of said tower, and removing a raffinate phase containing high viscosity index constituents from the top of the tower.

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