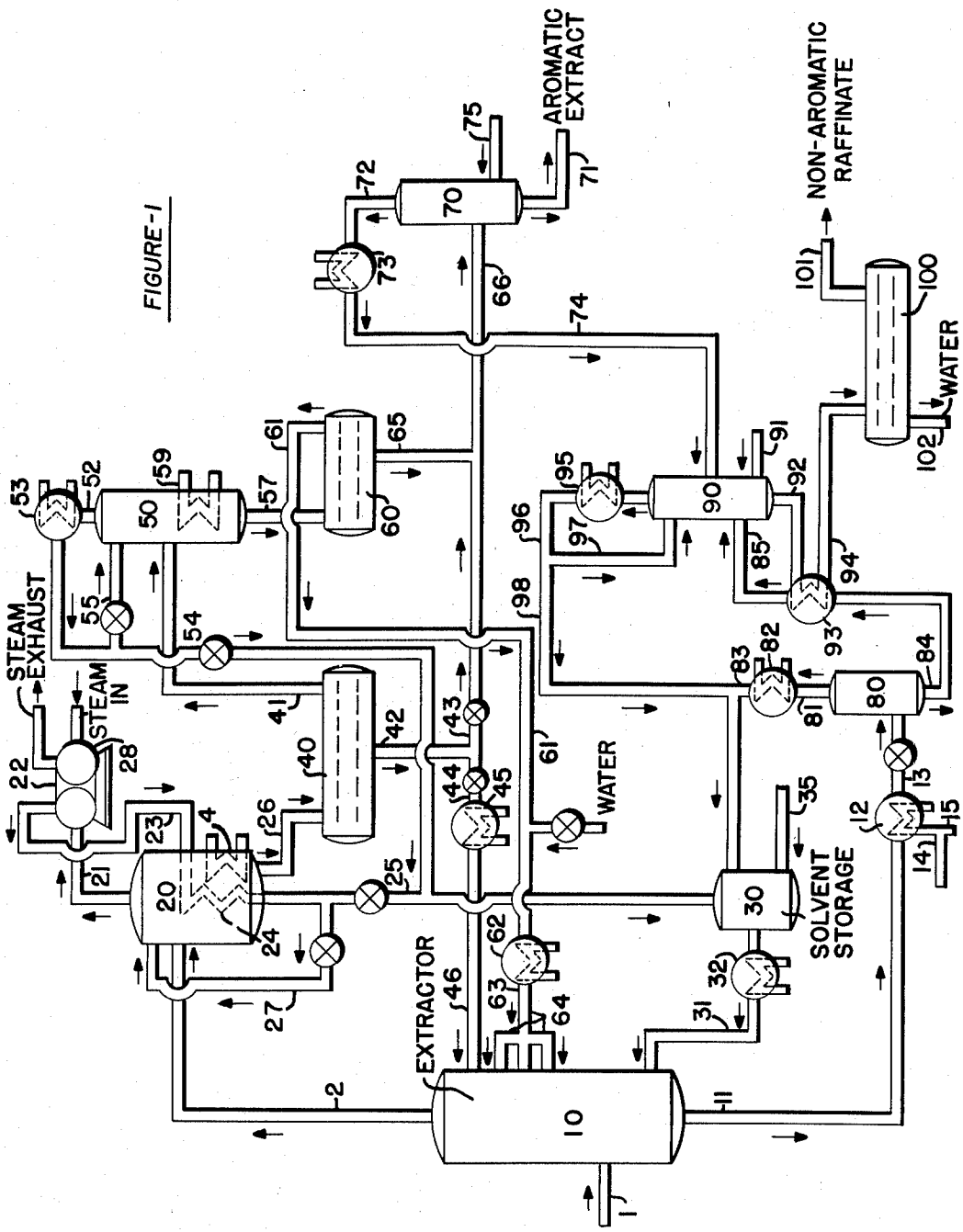
Nov. 15, 1955 M. R. FENSKE ET AL 2,723,940

SOLVENT EXTRACTION AND RECOVERY OF SOLVENT

Filed Nov. 12, 1952 2 Sheets-Sheet 1

FIGURE-1



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SOLVENT EXTRACTION AND RECOVERY OF SOLVENT

Filed Nov. 12, 1952 2 Sheets-Sheet 2

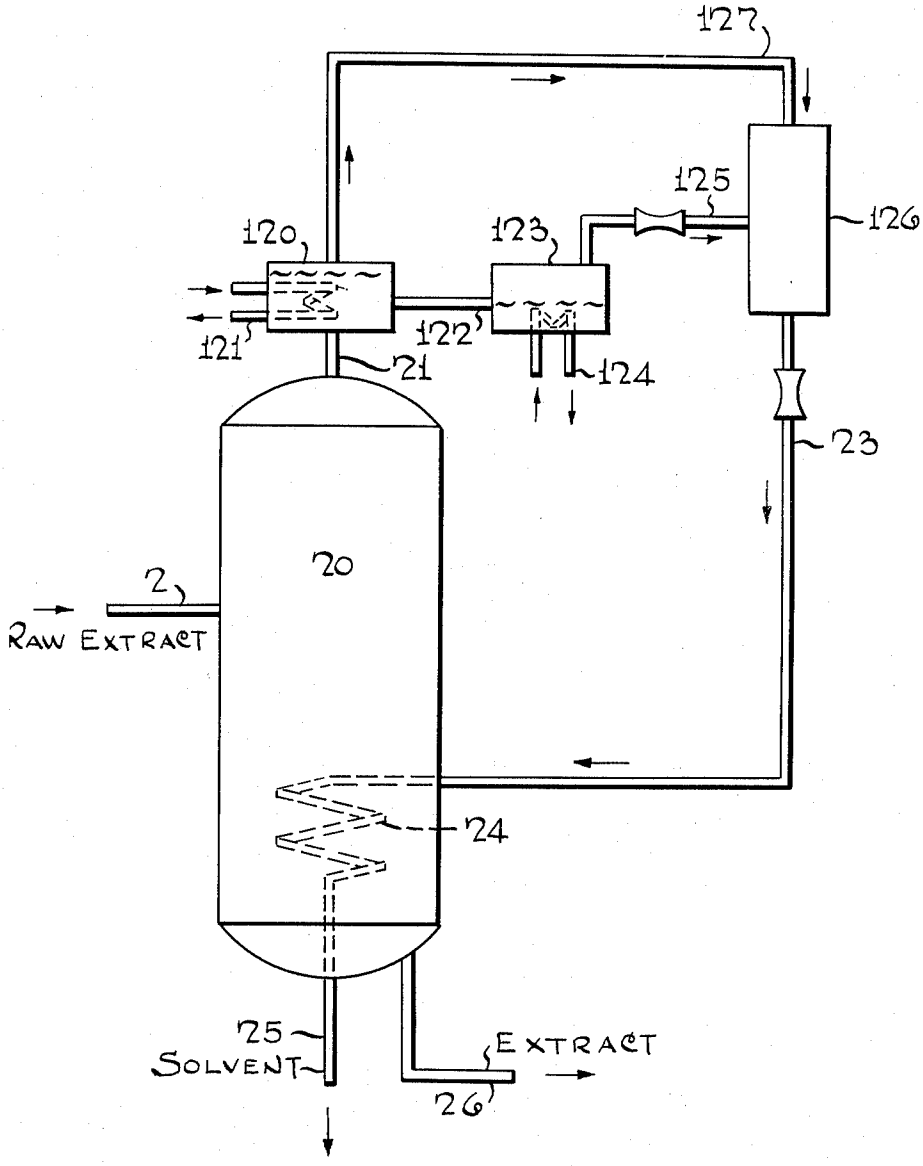


FIG. -2

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United States Patent Office 2,723,940
Patented Nov. 15, 1955

2,723,940

SOLVENT EXTRACTION AND RECOVERY OF SOLVENT

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Application November 12, 1952, Serial No. 320,016

14 Claims. (Cl. 196—13)

This invention relates to solvent extraction and recovery and regeneration of the solvent by compression distillation. More specifically it relates to the recovery of solvent in processes wherein aromatic or polar type compounds are extracted from a hydrocarbon stream. In one of its more specific aspects the invention relates to the extraction of aromatics from catalytically cracked cycle stocks with mixed ammonia solvents wherein water injection is resorted to for the purpose of controlling solubility and wherein the solvent is recovered by compression distillation.

It is well known that catalytically cracked cycle stocks usually contain large amounts of saturates as well as aromatics, the latter chiefly of the polynuclear type. These latter aromatics are deleterious in subsequent catalytic cracking. However, the saturates and non-aromatic portions represent a valuable source of additional cracking stock and would be upgraded in value over their present use in the ordinary cycle stock if they could be economically isolated.

Liquid extraction is an effective way to make this separation of saturates from aromatics or of the less polar types from the more polar or nucleated types. However, since a substantially pure solvent of properly controlled solvent power is normally essential for proper separation, solvent recovery costs and the cooling costs normally incident to solubility control in the extraction stage have heretofore greatly reduced the economic advantage of such separation. For instance, where a pure solvent may require about 5 theoretical extraction stages to produce a raffinate containing 10 weight percent aromatics and an extract containing 90 weight percent aromatics from a cycle oil containing 50 weight percent aromatics, the same separation would require 7 such stages if the solvent itself contained 2 weight percent aromatics under otherwise identical conditions, and with 3 percent aromatics in the solvent even an infinite number of stages would not make a raffinate containing only 10 percent aromatics.

It is the main object of the present invention to devise an improved extraction process. Another object is to reduce the solvent recovery costs of such a process without sacrificing the effectiveness of the separation. These objects, as well as the nature of the invention, will become more clearly apparent from the subsequent description and accompanying drawing.

In the drawing—

Figure 1 represents a schematic flow plan illustrating a suitable arrangement of equipment particularly adapted for the objects of this invention, using mechanical compression as a means for supplying the heat required in the distillation stage.

Figure 2 represents a similar flow plan wherein thermocompression is used instead of mechanical compression.

Unless indicated otherwise, it is to be understood that all percentages and ratios given herein are expressed on a weight basis.

In accordance with the present invention, an unusually effective and economical process for extraction of aromatics and highly polar compounds has been developed. In particular, it has been found that solvents which contain ammonia in mixture with certain other low molecular weight compounds are most satisfactory for extracting aromatics from mixed hydrocarbon streams. At the same time these solvents offer an unusual advantage in that, unlike heavier solvents such as phenol, furfural, or aniline, they can be recovered from the raw aromatic extract by compression distillation.

In other words, the solvents suitable herein are such that the solvent vapor produced in the evaporation of the raw aromatic extract can be appreciably compressed in an essentially adiabatic process without condensation. Thus a vapor stream is obtained which is materially hotter than the aromatic extract being distilled. This hot vapor can, therefore, be returnd to the distillation column and there passed in indirect heat exchange with the raw relatively cool extract solution. This causes the vapor to condense and the liberated latent heat is used to evaporate additional quantities of solvent from the raw extract. Thus, unlike in conventional distillation, the heat required for evaporation of the solvent is constantly being reclaimed and utilized within the process. At the same time no cooling water is needed for condensing the distillate since the extract being fed into the distillation column serves this purpose.

This independence on cooling water brings about another important advantage. Whereas conventional distillation normally must be operated at 120° F. or higher, that is, at a temperature at least 20 to 40° F. higher than the temperature of the available cooling water, a comparable compression distillation step is not limited in any such way. Hence compression distillation can be efficiently operated at 80° F. or lower.

The suitability of a solvent is determined by whether adiabatic compression of its saturated vapor produces a superheated (dry) vapor or a wet vapor. If a superheated vapor results, the solvent is operable. The production of a wet vapor on compression makes the solvent unsatisfactory. A test of applicability can be made by substituting in the following equation and solving for T_3.

$$\log \left(\frac{T_3}{T_1}\right)^{\frac{K}{K-1}} = \frac{\Delta H(T_2 - T_1)}{4.6(T_1)(T_2)}$$

T_1=saturation temperature at P_1, °K.
T_2=saturation temperature at P_2, °K.
T_3=temperature produced by adiabatic compression from T_1 and T_2 to P_2, °K.
ΔH=heat of vaporization, calories per mol.
K=ratio of molal specific heat at constant pressure to molal specific heat at constant volume.

If T_3 is higher than T_2, the solvent is applicable for use in our process. Such solvents include, among others, ammonia, methylamine, dimethylamine, ethylamine, propane, propylene, water, sulfur dioxide, carbon dioxide, methylchloride, and dichloro difluoromethane, and mixtures thereof. However, if T_3 is equal to T_2, an unsuitable wet vapor may be produced by compression. Normal butane, isobutane, phenol and furfural are examples of unsuitable materials. Usually solvents containing more than about 12 atoms per molecule are not suitable for use in our process. In addition to the foregoing characteristics, a suitable solvent also must have a normal boiling point within a practical range, for instance, between about −50 and +100° F.

Another feature of this invention is related directly to the extraction stage of the process. Normally an extraction stage comprises an extraction or stripping section and an enriching section. When using ammonia solvents in accordance with the present invention, the extraction section is generally located below the feed inlet and the enriching section thereabove. At constant temperature such as usually prevails in the extraction or stripping section, the solubility or weight percent of oil dissolved in the solvent phase increases as the solvent moves up the extractor toward the enriching section. This is due to the increasing aromaticity of the oil or hydrocarbon phase. However, in the enriching section it is desirable to maintain the solubility reasonably constant, since it has a great effect on the selectivity or relative solubility of the solvent. This is illustrated by the data of Table I which were obtained in extracting a typical cycle oil of about 40 percent aromatics content with a solvent composed of 45 percent methylamine and 55 percent ammonia.

Table I.—Effect of oil concentration on selectivity

Concentration of Oil in Solvent Phase, Percent	Selectivity, Beta [1]
10	20
20	6.5
30	2.0

[1] Exactly analogous to relative volatility "alpha" in distillation.

To counteract the reduction of selectivity due to increasing solubility, which in turn is due to the increasing aromaticity of the extracted oil, the temperature is progressively decreased in conventional enriching sections so as to keep solubility constant. For instance, to keep solubility constant at 25 percent, a temperature gradient of about 40° F. may be required between the oil feed stage and the top of or the end of the enriching section in a conventional extractor. If the oil feed stage is maintained at 100 to 120° F., it is therefore apparent that the amount of cooling required to produce this temperature gradient is large and is at a level which requires large quantities of cooling water as well as extensive heat transfer areas.

According to the preferred embodiment of this invention, solubility is controlled by use of an anti-solvent, rather than by temperature. For instance, the addition of 5 percent of water to an ammonia-amine mixture has been found to reduce the solubility of hydrocarbons therein to the same extent as lowering the temperature 25 to 30° F. Accordingly, depending on the nature of the hydrocarbon feed, solvent, and anti-solvent, up to 20 percent of anti-solvent can be injected as needed for solubility control. That is, the water or other anti-solvent is added to the solvent at two or more successive points in the upper part or enriching section of the extractor. While water is the preferred anti-solvent in the specific process described herein, any of the other solvents previously described may be used as an anti-solvent or solvent modifier for ammonia or for whichever other main solvent may have been chosen. Besides these, numerous other liquids, such as methanol, formamide, ethylene glycol and ethylene diamine, may be used as anti-solvents, since the anti-solvent normally remains in the liquid rather than in the vapor state, and hence need not possess the particular compressibility characteristics previously specified.

The water injection technique as proposed herein is particularly advantageous since its effect is to keep the solvent-to-oil ratio lower at the raffinate end of the extractor than at the extract end. This is due to the fact that the water introduced into the enriching section is in a solvent solution obtained as a bottoms product from the solvent recovery step. The use of a lower solvent-to-oil ratio at the raffinate end is helpful in that less of the extract solvent has to be evaporated. The distillation of a smaller amount of solvent effects a saving in steam in addition to a saving in heat transfer area required for the solvent evaporation. In fact the use of an increasing solvent-to-oil ratio resulting from the water injection is perhaps the ideal way to handle a separation on an equilibrium diagram such as is encountered here, that is, one in which the selectivity of the solvent decreases as the aromatic concentration increases. The reason is that the higher reflux ratio is applied in the region of low selectivity, which is where it is most needed. Note, for instance, that selectivity of the ammonia-amine solvent decreases from a value of about 6 when the oil stream contains 10% aromatics to a value of about 2 when the oil stream contains 90% aromatics.

EXAMPLE 1

The invention will now be described with reference to a specific example dealing with the extraction of a cracked cycle stock with an ammonia-amine solvent in a system illustrated in Figure 1 of the drawing. The feed is a catalytically cracked cycle oil boiling from 550 to 800° F. at atmospheric pressure and containing about 44% aromatics. Its gravity is 17.6° API. The solvent is a mixture of 40% ammonia and 60% monomethylamine. The ratio of solvent to feed oil is 2.7 at the raffinate end and 3.3 at the extract end, as described later. Water equivalent to 8% of the solvent is injected between the oil feed stage and the extract end to maintain solubility at 25% in the enriching section, that is, to maintain a saturated solution containing 25% of oil in solvent throughout the enriching section. The products are an extract and a raffinate containing 90 and 10% aromatics, respectively. The aromatics are recovered in a yield of 87% and a purity of 90%, while the non-aromatics are recovered in a yield of 92% and a purity of 90%. The compositions and flow rates of the various streams involved in the process below are summarized in Table II, wherein the numbers of the streams are keyed to Figure 1 of the drawing.

Referring to Figure 1, the cycle oil is fed into extractor **10** through line **1** at a temperature of 120° F. and a pressure of about 230 p. s. i. a. Ammonia-methylamine solvent at the same temperature and pressure is fed through line **31**. A liquid extract is removed through overhead line **2** and is composed of 75% solvent and 25% oil. The oil portion of the extract contains 90% aromatics. The extract at 120° F. and 230 p. s. i. a. is passed to compression distillation column **20** where solvent is boiled off at a pressure of 210 p. s. i. a. and a vapor temperature of 120° F., that is, substantially at extraction temperature and pressure. Enough solvent is taken from distillation unit **20** as vapor to produce a bottoms having an overall composition of 46 percent oil, 43 percent solvent, and 11 percent water. This solvent vapor contains about 45 percent ammonia and 55 percent methylamine. Substantially all of this vapor passes through line **21** to the suction side of a mechanical compressor **22**. This compressor advantageously is a rotary compressor driven by a steam turbine **28** which may be driven by steam at 1050° F. and 500 p. s. i. a. However, other types of compressors may be used.

Table II.—Key to Fig. 1

[Solvent-to-oil ratio=2.7 to 1 by wt. at raffinate end; solvent composition=60 wt. percent monomethylamine+40 wt. percent ammonia; 8 wt. percent water injected into solvent between oil feed stage and extract end; aromatic yield=87%; aromatic purity=90%; non-aromatic yield=92%; non-aromatic purity=90%]

Stream		Wt. percent Oil	Wt. percent Solvent	Wt. percent Aromatics in Oil	Wt. percent Ammonia In Solvent	Temp., ° F.	Pressure, p. s. i. a.	Flow, 1,000 lbs. Per Hour			
Numeral	Composition							Solvent	Oil	Water	Total
1	Oil Feed	100	0	44		120	230	0	66.6	0	66.6
2	Extract	25	75	90	40	120	230	200.0	72.6	17.4	290.0
11	Raffinate	83	17	10	30	120	230	7.8	38.3	0	46.1
13	do	83	17	10	30	210	150	7.8	38.3	0	46.1
14	Steam from Turbine					280	15	0	0	2.1	2.1
15	Steam					280	15	0	0	1.9	1.9
21	Solvent Vapor	0	100		45	120	210	132.1	0	0	132.1
23	do	0	100		45		400	132.1	0	0	132.1
25	Liquid Solvent	0	100		45	165	400	132.1	0	0	132.1
26	Bottoms	46	43	90	25	145	210	67.9	72.6	17.4	157.9
29	Steam to Turbine	0	0			1,050	500	0	0	18.4	18.4
31	Solvent Feed	0	100		40	120	230	179.8	0	0	179.8
35	Makeup Solvent										
41	Solvent Layer	25	56	90	25	145	210	51.3	22.9	17.4	91.6
42	Oil Layer	75	25	90	25	145	210	16.6	49.7	0	66.3
44	Reflux Oil	75	25	90	25	145	210	14.2	42.6	0	56.8
46	do	75	25	90	25	120	210	14.2	42.6	0	56.8
52	Solvent Vapor	0	100		30	120	170	32.8	0	0	32.8
54	Liquid Solvent	0	100		30	120	170	32.8	0	0	32.8
57	Bottoms	39	31	90	20	190	170	18.5	22.9	17.4	58.8
59	Steam from Turbine					280	15	0	0	10.3	10.3
61	Water Layer	5	48	90	20	190	170	17.4	1.7	17.4	36.5
64	do	5	48	90	20	120	170	17.4	1.7	17.4	36.5
65	Oil Layer	95	5	90	20	190	170	1.1	21.2	0	22.3
66	Extract Product	89	11	90	20	175	170	3.5	28.3	0	31.8
71	do	100	<0.1	90		210	15	0	28.3	0	28.3
74	Solvent+Water					120	15	3.5	0	6.0	9.5
75	Steam from Turbine					280	15	0	0	6.0	6.0
81	Solvent Vapor	0	100		40	210	150	4.9	0	0	4.9
83	Liquid Solvent	0	100		40	210	150	4.9	0	0	4.9
84	Flashed Raffinate	93	7	10	30	210	150	2.9	38.3	0	41.2
85	do	93	7	10	30	340	150	2.9	38.3	0	41.2
91	Steam					360	150	0	0	4.3	4.3
92	Raffinate+Water			10		360	150	0	38.3	10.3	48.6
94	do			10		210	150	0	38.3	10.3	48.6
96	Liquid Solvent	0	100		30	120	150	9.6	0	0	9.6
97	Reflux Solvent	0	100		30	120	150	3.2	0	0	3.2
98	Liquid Solvent	0	100		30	120	150	6.4	0	0	6.4
101	Raffinate Product	100	<0.1	10		210	150	0	38.3	0	38.3
102	Water					210	150	0	0	10.3	10.3

In compressor **22** the solvent vapor is compressed to a pressure of about 400 p. s. i. a., whereby it is superheated to about 180° F. The compressed superheated vapor then passes through line **23** to reboiler **24** which is positioned in the bottom part of distillation tower **20**. Thus the superheated vapor is cooled to a saturation temperature of about 165° F., at which point it is condensed with the liberation of heat. This heat is used for boiling off additional solvent in tower **20**. The condensed vapor stream **25** is passed to storage tank **30** for reuse. A small amount of condensate may also be returned through line **27** to serve as reflux in tower **20**, to improve the purity of the solvent distillate stream **21**.

An auxiliary heat source such as steam coil **4** is provided to heat the initial raw extract to its boiling point. After that the compressor **22** is started and supplies further heat by mechanical means, whereas the auxiliary heat source is then cut out.

The bottoms from tower **20** contain 46% oil, 43% solvent, and 11% water. They have a boiling point of about 145° F. and form a two-phase emulsion at that temperature. A bottoms stream **26** is, therefore, taken to settler **40** for phase separation. The top or solvent layer will contain 25% oil, 56% solvent, and 19% water. The bottom layer will contain about 75% oil and 25% solvent.

The solvent layer is passed through line **41** to a conventional distillation tower **50** for further solvent removal. Sufficient solvent is removed in tower **50** to produce a bottoms stream **57** having an overall composition of about 39% oil, 31% solvent and 30% water. These bottoms exist as two liquid phases at their boiling point, 190° F. Thus after passage to settler **60**, the bottoms separate into a water layer consisting of 48% solvent, 5% oil, and 47% water, and an oil layer containing 5% solvent and 95% oil. A solvent vapor stream **52** containing about 30% ammonia and 70% methylamine is removed from tower **50** and passed through water condenser **53**. The resulting liquid solvent stream **54** is returned to storage tank **30**. A small or appropriate amount of condensed solvent may be returned through line **55** as a reflux for tower **50**. Heat for column **50** may be supplied by introducing steam through line **59**. Since these bottoms boil at about 190° F., atmospheric exhaust steam from turbine **28** can be used since such steam has a temperature of 280° F.

While more solvent could be removed in compression distillation unit **20**, this would materially increase the boiling point of the bottoms **26**. Consequently, the vapor **21** would have to be compressed more to provide the required temperature differential across the reboiler surface, and this would usually reduce the outstanding advantages of compression distillation because of the increased work of compression and also because of the higher pressure equipment required. The amount of solvent removed in the compression distillation unit is usually fixed so that the temperature differential between the boiling bottoms and the condensing compressed vapor is at least 20° F. when an adiabatic compression ratio of about 2 to 1 is used.

The water phase from separator **60** is recycled through line **61**. After cooling to about 120° F. in cooler **62**, it is introduced through line **63** into the enriching section of extractor **10** at three or more injection points **64** located between the oil feed stage and the extract end. The amount of water thus injected into extractor **10** amounts to about 8% based on the solvent. The anti-solvent effect of this water serves to maintain the solu-

bility of the oil in the solvent phase in extractor **10** at the desired level.

The required water is added to the system at the start and thereafter operates in a closed cycle. Makeup water can be pumped into stream **61** as needed.

The oil phase consisting of about 95% oil and 5% solvent is withdrawn from separator **60** through line **65** and combined with oil stream **43**, which is part of the heavy phase **42** withdrawn from separator **40**. Stream **43**, which consists of about 75% oil and 25% solvent is sufficient in amount to produce the desired amount of aromatic extract product when combined with stream **65**. The remainder of the heavy phase **42** from separator **40** is passed through line **44**. After cooling to 120° F. in water cooler **45**, it is returned through line **46** to the extract end of extractor **10** as reflux.

The combined product in line **66** typically may consist of about 89% oil and 11% solvent, with only a trace of water. The combined product stream **66** is sent to tower **70** for final solvent removal.

It will be noted that solvent feed stream **31**, reflux stream **44**, and water recycle stream **61**, all have to be cooled to extraction temperature prior to their introduction into extractor **10**. In the example this cooling is accomplished by heat exchange with cooling water in coolers **32**, **45** and **62**, respectively. However, the required cooling can be obtained in other ways. For instance, a very advantageous simplification of equipment can be achieved by flashing all three streams at a low pressure which results in autorefrigeration. All of the resulting solvent vapor can then be condensed in condenser **73** which operates at atmospheric pressure.

The extract stream **66** entering tower **70** is flashed to atmospheric pressure and stripped with open steam introduced through line **75**. The amount of steam used for stripping is preferably at least that which will produce as top product a mixture composed of 40% solvent and 60% steam. Such a mixture can be condensed at atmospheric pressure with cooling water at 100 to 120° F. The steam used here can also be a portion of the exhaust from turbine **28**. The aromatic oil product stream **71** leaving tower **70** as bottoms will have less than 0.5, and preferably less than 0.1 per cent solvent.

The essentially non-aromatic raffinate leaves the bottoms of extractor **10** through line **11** at 230 p. s. i. a. and 120° F. It contains 17% solvent and only a trace of water. This raffinate stream is flashed at 150 p. s. i. a. and 210° F. in tower **80**. The sensible and latent heat required for the flash may be supplid in heat exchanger **12** by steam **14** which may be at least in part exhaust steam from turbine **28**. Additional extraneous steam may be supplied through line **15**. The resulting solvent vapor stream **81** is condensed in condenser **82** and returned to storage tank **30** through line **83**.

The raffinate bottoms pass through lines **84** and **85** to finishing tower **90**. This tower strips the remaining solvent from the raffinate at 360° F. with open steam introduced at **91**. Tower **90** operating at 150 p. s. i. a. produces pure, water-free solvent as the distillate. The raffinate and water are taken as bottoms at about 360° F. through lines **92** and **94** and separated from each other in separator **100**. The hot bottoms from tower **90** may be indirectly contacted in a heat exchanger **93** with the crude raffinate stream **84** to preheat the latter prior to flashing in tower **90**. The water settled out in separator **100** is discarded through line **102** while the raffinate product is drawn off through line **101**. This raffinate contains only 10% aromatics and represents a superior stock for catalytic cracking.

Tower **90** is also advantageously used to recover the solvent from the mixed solvent-water vapors produced in tower **70**. For this purpose the distillate stream **72**, after condensation in atmospheric condenser **73**, is passed through line **74** to tower **90**. The pure solvent vapor from tower **90** is condensed in condenser **95**. A portion

of the condensate stream **96** is returned through line **97** to tower **90** as reflux while the remainder is returned through line **98** to storage tank **30**. Make-up solvent may be added through line **35**.

The total hourly steam requirement for the illustrated process is as follows:

Steam	Lbs. per Hour
1050° F. at 500 p. s. i. a.	[1] 18,400
360° F. at 150 p. s. i. a.	[2] 4,300
210° F. at 15 p. s. i. a.	[3] 1,900
	24,600

[1] For turbine 28; exhaust used in towers 50 and 75, and in heater 12.
[2] For raffinate stripper 90.
[3] Additional steam for raffinate heater 12.

The above total of about 25,000 pounds of steam per hour compares with about 80,000 pounds of steam per hour required for solvent recovery when conventional distillation is used instead of the described compression distillation in an otherwise similar system. Compression distillation, of course, requires somewhat higher grade steam than conventional distillation. In addition to this saving on the weight of steam, compression distillation saves cooling water. Finally, as also pointed out earlier herein, a major amount of cooling is also saved in the present invention by water injection, which makes it possible to control solubility in the extractor without having to rely on any temperature differential. It is thus clear that solvent extraction systems embodying compression distillation have major practical advantages in commercial operations, particularly when water injection is also used. The data show that water injection does not add to the steam requirements, nor does it make the solvent recovery more complex.

While the solvent employed in the example consisted of 40% ammonia and 60% monomethylamine, solvents containing the same ingredients in different proportions may often be preferred. Such changes in solvent composition can be used to adjust the solubility of the solvent to the desired value within a wide range of temperatures. This in turn allows considerable freedom in choosing the temperature at which the extraction is performed. For instance, a solvent containing 35% of the amine and 65% ammonia will produce 10% solubility at 160° F. when in equilibrium with a catalytic cycle oil containing 10% aromatics. In comparison, the same solubility is obtained at 120° F. if the solvent contains 60% of the amine and 40% ammonia. This equivalence of the effect of 25% amine concentration and 40° F. in temperature level has been found to be generally applicable to hydrocarbon extraction work.

The solvent composition, temperature, and water injection ratio are the variables which can be changed to produce the desired solubilities in the extractor. The pressure on the extractor is fixed to maintain the solvent in the liquid phase.

The top temperature of the compression distillation tower should be at least as high as the entering feed stream. The pressure on the tower is determined so that the vapor at the top of the tower is saturated. The temperature of the bottoms is fixed so that a compression ratio of about 2 to 1 will produce a temperature differential of 20° F. or more across the heating surface of the reboiler.

The pressure on towers **50**, **70**, **80** and **90** is such that cooling water at 100° to 120° F. can be used in the condensers.

EXAMPLE 2

While the above example related principally to the upgrading of a catalytic cycle stock into a high-quality feed stock for catalytic cracking by removal of aromatics therefrom, the invention may be similarly applied to other feed stocks and for other purposes. For instance, the

present invention may be useful for separating naphthas into a largely non-aromatic raffinate and an extract rich in aromatics and consequently characterized by an improved octane number.

A thermally reformed, or cracked, naphtha boiling between about 130 and 400° F., and containing 25 vol. per cent olefins, 25 vol. per cent aromatics, and 50 vol. per cent saturates, with a research octane number of 71, was extracted at about 75° F. with a solvent composed of 80 wt. per cent ammonia and 20 wt. per cent monomethylamine. The extract portion, comprising 50% of the original naphtha, had a clear research octane number of 97, while the raffinate rated 46 octane number. Over 80% of the extract was composed of aromatics and olefins.

Since lower molecular weight hydrocarbons are generally more soluble, the solvent contains more ammonia than that used in the previous example relating to catalytic cycle stock. The use of a higher ammonia concentration in the solvent would increase somewhat the pressures throughout the process.

The process as described by the drawing is also operable for the case of naphtha extractions. An even simpler process, utilizing the same principles, can be advantageously used. Towers **50** and **70** and separators **40** and **60** in the extract solvent recovery system would be deleted. The bottoms from tower **20** would pass to a duplicate of tower **90**. Open steam would strip the solvent out of the extract hydrocarbon. Pure solvent vapor would be the top product and a mixture of hydrocarbon and water would be the bottoms. The bottoms would pass to a phase separator wherein liquid water is separated from the liquid hydrocarbon. Enough of the hydrocarbon layer to satisfy the material balance would be taken as extract product. The remainder of the hydrocarbon layer would be sent back to the extractor as reflux. The water required for injection into the extractor would be taken from the water layer in the phase separator. The remaining water would be discarded. By increasing the compression ratio in the compression distillation step, thus allowing more solvent to be removed in that step, the economics would be as good as those of the previous example.

EXAMPLE 3

Instead of distilling the solvent with the aid of mechanical compression as illustrated in Fig. 1, thermocompression may be used. Thermocompression expands high pressure vapor through a nozzle to obtain a high velocity gas. This high velocity gas then draws low pressure vapor into a mixing zone and the mixture is compressed by a second nozzle as hereafter described.

Referring to Fig. 2 which illustrates a typical embodiment of the thermocompression scheme, the extract stream **2** from the extractor (not shown) may be fed to distillation tower **20** as a liquid at 80° F. The solvent stream **21** vaporized in tower **20** may be withdrawn at 80° F. and 150 p. s. i. a. and passed to a condenser **120**. There a portion of the solvent vapor is condensed with the aid of cooling coil **121** or equivalent cooling means, preferably in equilibrium with the uncondensed vapor so that the temperature of the liquefied solvent is not reduced appreciably below its condensation point. The vapor not condensed in condenser **120** is withdrawn through line **127** and serves as the low pressure ammonia for the thermocompression ejector **126**. The liquefied solvent is passed from condenser **120** through line **122** to an ammonia boiler **123**. High pressure ammonia vapor needed by the ejector is produced in boiler **123** with the aid of a steam coil **124** or the like. The mixed superheated vapor stream **23** leaving the ejector **126** has a pressure intermediate to that of the high and low pressure streams. The pressure of solvent vapor **23** preferably corresponds to a saturation temperature about 20° F. above the boiling point of the bottoms in tower **20**, inasmuch as condensation of stream **23** in reboiler **24** serves to supply the heat required for evaporation of additional ammonia solvent from the extract phase being fed into tower **20**. Thus, in the scheme described it may be desired that stream **23** have a temperature of about 160° F. and a pressure of 286 p. s. i. a. The resulting condensed solvent **25** and the concentrated raw extract **26** may thereafter be treated substantially as described earlier in connection with Figure 1.

The actual temperatures and pressures of the various streams in the thermocompression step may of course vary considerably depending upon the temperature level of tower **20**, on the ratio in which it is desired to combine the high and low pressure streams in the ejector-type jet pump, as well as on the overall efficiency of the thermocompression step. This latter may advantageously be in the range of about 0.75 to 0.90 and depends on the efficiency of expansion, compression, and transfer of momentum. Characteristic values for these three types of efficiencies may be 0.98, 0.95, and 0.85, respectively. The weight ratio of condensed to uncondensed solvent leaving condenser **120** may be between about 2:1 and 1:3. As a result, for instance, when the weight ratio of high and low pressure ammonia in the ejector is 1:1, it is desirable to raise the ammonia in steam boiler **123** to a temperature of about 290° F. and a pressure of about 710 p. s. i. a. In this arrangement about 3.1 pounds of ammonia are evaporated with steam per pound of steam required by the steam boiler, as compared with about 2 pounds of evaporation per pound of steam in conventional distillation. At the same time, while not quite so efficient, thermocompression has an advantage over mechanical compression since it requires lower grade steam and no mechanical compressor. Furthermore, when the ratio of high to low pressure ammonia in the ejector is reduced to 1:2, then 3.9 pounds of ammonia can be evaporated per pound of steam. However, the additional economy is accomplished by the use of high pressure ammonia at about 400° F. and 1500 p. s. i. a. as opposed to the 290° F. and 710 p. s. i. ammonia when a 1:1 ratio is used.

A further advantage can be obtained by combining thermocompression with compression distillation. In the above described thermocompression scheme some heat is wasted in the preparation of high pressure ammonia, since ammonia gas is first condensed, only to be subsequently reheated in the steam boiler. Instead of this, it is possible to replace the condenser **120** and steam boiler **123**, shown in Fig. 2, by a mechanical compressor, e. g. a steam-driven centrifugal compressor. In such a case, when the ejector **126** is operated at a 1:1 ratio of high to low pressure ammonia, stream **21** is simply split in half, one-half of the vapor passing directly to the ejector whereas the other half is mechanically compressed to 710 p. s. i. a. and a temperature of 290° F. prior to entering the ejector. When this is done by a centrifugal compressor driven by steam at 1050° F. and 500 p. s. i. a., 0.31 pound of steam is required to compress a pound of ammonia vapor to the desired pressure, assuming that the steam expands to atmospheric pressure and that the efficiency of the turbine and compressor is 90 and 80 percent, respectively. Since two pounds of solvent are evaporated in tower 20 per pound of high pressure ammonia in line **125**, about 6.5 pounds of ammonia solvent are evaporated per pound of steam. Again, the efficiency is not so high as if the entire compression is done mechanically as shown in Fig. 1. However, only one-half the amount of material has to be compressed in this combined mechanical-thermocompression case.

Still other modifications and variations of the invention may become apparent to those skilled in the art without departing from the scope and spirit of the appended claims.

We claim:

1. In a process for recovering a solvent from a liquid extract solution containing the solvent substantially sat-

urated with a hydrocarbon oil having an atmospheric boiling point between about 175 and 1000° F., said solvent being capable of being compressed adiabatically from a saturated vapor to a superheated vapor, the improvement which comprises passing the substantially saturated extract solution from an extraction zone substantially at extraction temperature and pressure to a distillation zone, evaporating solvent from said saturated solution in the distillation zone substantially at said extraction temperature and pressure so that the resulting residual liquid becomes a two-phase system containing a solvent-rich phase and an oil-rich extract phase, withdrawing the resulting solvent vapor overhead from the distillation zone, compressing substantially all of the withdrawn solvent vapor sufficiently to heat it at least 20° F. above the temperature of said residual liquid present in the distillation zone, passing the compressed hot solvent vapor in indirect heat exchange with the liquid present in the distillation zone, whereby the compressed solvent vapor is condensed and additional solvent is evaporated from the extract solution, recovering the condensed solvent, withdrawing said residual two-phase liquid from the distillation zone, and mechanically separating said residual liquid into a solvent-rich phase and an oil-rich extract phase.

2. A process according to claim 1 wherein the solvent contains from 20 to 100% ammonia.

3. A process according to claim 1 wherein the solvent is a mixture of 20 to 70% ammonia and 80 to 30% monomethylamine.

4. A process according to claim 3 wherein the hydrocarbon is a catalytically cracked cycle oil boiling between about 400 and 1000° F. at atmospheric pressure and containing about 30 to 60% of aromatic hydrocarbons.

5. In a process for recovering a solvent composed of 40 to 65% methylamine and 60 to 35% ammonia from a liquid extract solution comprising about 60 to 90 parts of solvent, substantially saturated with 40 to 10 parts of a petroleum cycle oil fraction, the improvement which comprises passing the solution under pressure into a distillation zone where the solution is maintained at its boiling point evaporating enough solvent so that the residual liquid becomes a two-phase system containing a solvent-rich phase and an oil-rich extract phase, withdrawing an overhead stream of saturated solvent vapor from the distillation zone, compressing substantially all of the withdrawn solvent vapor sufficiently to heat it at least 20° F. above the aforesaid boiling point, passing the compressed hot solvent vapor in indirect heat exchange with the solution present in the distillation zone, recovering the resulting condensed solvent, withdrawing said two-phase liquid from the distillation zone, and separating said solvent-rich liquid phase from said oil-rich liquid extract phase.

6. A process according to claim 5 wherein the entire stream of saturated solvent vapor withdrawn from the distillation zone is compressed mechanically to a pressure corresponding to a saturation temperature at least about 20° F. above the boiling point of the said extract solution.

7. A process according to claim 5 wherein the saturated solvent vapor withdrawn from the distillation zone is separated into an ejector stream and a direct stream in a weight ratio ranging between about 2:1 and 1:3, the ejector stream is compressed to an elevated pressure and a superheated condition, and the two vapor streams are mixed to produce a superheated mixed vapor having a pressure corresponding to a saturation temperature at least about 20° F. above the boiling point of the said extract solution.

8. A process according to claim 7 wherein the ejector stream is compressed mechanically.

9. A process according to claim 5 which comprises the specific steps of withdrawing the saturated solvent vapor from the distillation zone at a temperature of about 80° F. and about 150 p. s. i. a., cooling the said vapor in a condensation zone so as to condense about ½ to ⅓ of the said solvent vapor, withdrawing the condensed solvent from the condensation zone and heating it to produce a superheated vapor at a temperature of about 290 to 400° F. and a pressure of about 700 to 1500 p. s. i. a., and mixing the superheated vapor with uncondensed portion of the solvent vapor to produce a superheated mixed vapor having a pressure corresponding to a saturation temperature at least about 20° F. above the boiling point of said extract solution.

10. A process according to claim 5 wherein said separated liquid extract phase hydrocarbon bottoms is passed to a stripping zone where additional solvent is stripped from the said extract phase at relatively low pressure.

11. A process for separating aromatic hydrocarbons from a cycle oil which comprises passing the cycle oil into an intermediate part of an extraction zone at an extraction temperature of about 70 to 170° F., introducing a liquid ammonia solvent substantially at extraction temperature into a lower part of the extraction zone, maintaining the extraction zone under sufficient pressure to keep the solvent mixture in liquid phase, withdrawing a raffinate stream containing an oil of less than 20% aromatics content from the bottom part of the extraction zone, withdrawing under pressure an extract stream containing liquid solvent substantially saturated with oil of at least 75% aromatics content from the upper part of the extraction zone, passing the extract stream into a distillation zone maintained at substantially said extraction temperature and at a pressure permitting evaporation the extract stream, removing overhead the resulting solvent vapor so as to form a two-phase liquid containing an oil-rich extract bottoms phase and a solvent-rich bottoms phase in the bottom of said distillation zone, superheating substantially all of the withdrawn solvent vapor by mechanical compression to a temperature at least 20° F. higher than the aforesaid boiling point, passing the superheated solvent vapor in indirect heat exchange with the liquid extract present in the distillation zone, recovering the resulting condensed solvent for reuse, withdrawing said two-phase liquid from the distillation zone, separating said solvent-rich bottoms phase from said oil-rich extract bottoms phase, stripping the separated oil-rich extract phase in an extract stripping zone with steam at substantially atmospheric pressure to strip out additional solvent and to produce a concentrated extract rich in aromatics and containing less than 0.5% solvent, recovering the flashed off solvent, and recovering said concentrated aromatic extract.

12. A process according to claim 11 wherein the raffinate is steam stripped in a raffinate stripping zone at a pressure sufficient to maintain water in liquid phase, the overhead vapors from the extract stripping zone are pumped into the raffinate stripping zone, a solvent vapor overhead is recovered from the raffinate stripping zone, and a bottoms cut containing an essentially solvent-free oil and water is also recovered from the raffinate stripping zone.

13. A process according to claim 11 wherein the solvent comprises a mixture of ammonia and methylamine, the extraction zone is maintained throughout at uniform temperature, a water stream is injected into the upper part of the extraction zone at a plurality of points to maintain the oil solubility substantially constant above the feed inlet, the solvent phase separated from the liquid bottoms of the distillation zone is further distilled in a secondary distillation zone, additional solvent is recovered overhead from the secondary distillation zone, a bottoms stream is also withdrawn from the secondary distillation zone and separated into a water phase and a concentrated aromatic extract phase, the water phase being recycled for injec-

tion into an enriching section of the extraction zone, and the aromatic extract phase is recovered.

14. In a process according to claim 11 wherein solvent is recovered from a substantially saturated extract stream, the further improvement which comprises returning a portion of the separated oil-rich liquid extract bottoms phase as reflux to an enriching section of the extraction zone from which the first mentioned liquid extract stream is being obtained for solvent recovery.

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