

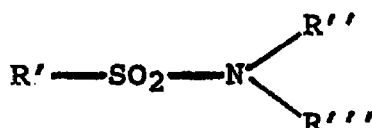
Description

This invention is directed at an improved extractive separation process. More specifically, the present invention is directed at an improved method for directing feed and/or recycle streams into an extraction zone to improve the extraction zone performance.

In the separation of a mixture of components, one or more unit operations may be utilized. The particular unit operation normally is selected after a consideration of several variables, including the desired purity of the separated fractions, capital equipment cost, equipment reliability and operating costs. In many separation operations, extractive separation may be the preferred method of separation. Typically, a solvent having the desired affinity for one or more of the feed and/or recycle stream components is passed through the extraction zone counter-currently to the feed and/or recycle streams to effect the separation. For example, in the separation of aromatics, such as benzene, toluene, and xylene (BTX) from non-aromatics, such as hexane, heptane, and isoheptane, the feed and/or recycle stream may be contacted with a solvent such as sulfolane (tetrahydrothiophene-1,1-dioxide), tetraethylene glycol, phenol, dimethylsulfoxide, furfural, or N-methyl pyrrolidone. In the preparation of a lube oil basestock from a hydrocarbon feedstock the hydrocarbon feedstock may be extracted with a solvent such as phenol, N-methyl pyrrolidone and furfural.

In the conventional process, the solvent typically is introduced substantially at the top of the extraction zone, flows downwardly, and exits at the bottom enriched with the extracted component. The hydrocarbon inlet feedstream typically is introduced at a single location at or near the bottom of the extraction zone, flows upwardly, and exits near the top of the extraction zone with a reduced concentration of the extracted component. The inlet recycle stream, if present, typically is introduced at a single location below the hydrocarbon feed location to achieve a backwashing effect for displacing certain components from the solvent before the solvent exits from the base of the extractor. In the extractive separation of aromatics from non-aromatics, recycle stream components may displace from the aromatic-rich solvent into the raffinate certain non-aromatic components which would be more difficult to remove from the BTX product in downstream purification steps. This recycle stream typically may be a product stream from a downstream purification stage which removes non-aromatics from the aromatic products.

EP-A-25765, equivalent to US-A-4316796, describes and claims a process for the solvent extraction of dienic and/or aromatic hydrocarbons present in hydrocarbon fractions employing liquid-liquid extraction and/or extractive distillation, said process being characterized in that the solvent comprises at least one sulfonamide of the general formula



wherein R', R'' and R''' can be linear or branched, saturated or unsaturated, aliphatic groups possessing from 1 to 18 carbon atoms, wherein two or three of groups R', R'' and R''' can be identical, wherein one of the groups R'' or R''' can be replaced with a hydrogen atom, and wherein at least one of groups R', R'' and R''' is unsaturated. EP-A-25765 describes and illustrates (Figure 2) a solvent extraction process wherein there is a recycle line for the return of all (i.e. 100%) recovered and recycled solvent to the solvent extraction column at a location between the feed introduction point and the solvent addition points. There is no disclosure or suggestion in EP-A-25765 that only some of the solvent should be recycled to the said location.

US-A-3065169 describes and claims a process for the extraction and recovery of aromatic hydrocarbons from a liquid hydrocarbon mixture containing aromatic and non-aromatic hydrocarbons comprising (1) introducing the mixture into a counterflow multi-stage extraction system wherein the temperature is maintained between 130 and 155°C; (2) introducing, at one end of the extraction system, a high boiling, aromatic selective solvent having a normal boiling temperature within the range of 225°C to 295°C and which solvent contains dissolved water in the proportion of not more than 2% by weight; (3) passing the solvent in counter-current flow to the hydrocarbon mixture to obtain a solvent extract phase enriched in aromatics; (4) withdrawing the extract phase from the extraction system and introducing the said extract phase at a temperature between 125° and 155°C without appreciable flash vaporization into a stripping zone, which is operated at a pressure of at least 1.5 atmospheres absolute and at a top temperature

between 125° C and 155° C and at a bottom temperature of at least 165° C to separate overhead a vapor phase enriched in non-aromatic hydrocarbons; (5) condensing the vapors from said zone and forming a first liquid phase consisting essentially of hydrocarbons along with a second liquid phase and recycling them to the extraction system after removing substantially all water present as the second liquid phase, with the condensed vapors being introduced into the extraction system between the intermediate point of the system and the end at which the extract phase is withdrawn; (6) withdrawing the liquid remainder of the extract phase from the stripping zone and separating the solvent from the hydrocarbons thereof in a distilling zone at a pressure of less than 0.5 atmospheres absolute, said zone having a bottom temperature of at least 145° C, which is at least 10° C lower than the bottom temperature in the stripping zone; and (7) withdrawing the stripped solvent from the distilling zone, cooling said stripped solvent by at least 15° C to a temperature between 130° and 155° C, and returning the cooled solvent to the extraction system.

In the process of US-A-3065169, a recycled stream of recovered solvent is introduced into the extraction zone at the end region thereof from which the aromatic rich extract is withdrawn. There is no disclosure or suggestion that the recycled stream should be introduced into the extraction zone above the point of introduction of the feedstream.

In many extractive operations, it may be desired to improve the capacity and/or separation achieved in the extraction zone of new or existing extractive separation zones without the need for additional processing equipment and without increasing the size and/or operating costs. In other operations, upstream modifications or changes in feeds may have altered the composition or flow rates to the extraction zone from those utilized in the original design of the extraction zone.

Accordingly, it is desirable to provide a reliable, flexible process which may even be retrofitted to existing extraction zones to permit increased throughput rates without adversely affecting product quality and without causing a significant increase in operating costs.

It is also desirable to provide a process which may improve the feed separation in an extraction zone.

The subject invention defined below, is based upon the discovery that the capacity and/or separation efficiency of an extractive separation in which a recycle stream from a downstream product purification step is recycled to the extraction zone may be improved if portion of the recycle is added to the extraction zone at a location above the location at which all the feed is added to the extraction zone. This improvement in extraction zone efficiency permits an increase in product quality or throughput.

The present invention provides a method for the extractive separation of an inlet feed stream comprising first and second components into raffinate and extract streams, comprising :

- (a) introducing the inlet feed stream into an extraction zone;
- (b) introducing a solvent selectively miscible with one of the components into the extraction zone at a spaced-apart location from the location at which the feed stream is introduced;
- (c) causing the feed stream and solvent stream to pass substantially countercurrently through the extraction zone to produce an extract product stream relatively rich in the first component and a raffinate product stream relatively rich in the second component; and
- (d) returning a separate recycle stream, produced by subsequent processing of at least one of the extraction zone product streams, into the extraction zone; characterised by:
- (e) introducing a portion of the recycle stream containing substantially all of the second component of the extract product stream into the extraction zone at a location between the locations at which the solvent and all the feed stream are introduced into the extraction zone.

The first component may be an aromatic component and the second component may be a non-aromatic component.

In a preferred embodiment the first component comprises an aromatic selected from the group consisting of benzene, toluene and xylene and mixtures thereof. The second component preferably comprises a non-aromatic. The solvent preferably is selectively miscible with the aromatic component and is selected from the group consisting of sulfolane, tetraethylene glycol, phenol, dimethylsulfoxide, furfural, N-methyl pyrrolidone and mixtures thereof. Preferably at least 5 wt.% of the recycle stream and more preferably at least 25 wt.% of the recycle stream is added to the extraction zone at a location above the location at which all the feed is added to the extraction zone.

BRIEF DESCRIPTION OF THE DRAWING

The Figure is a simplified schematic flow drawing to illustrate various methods of solvent extraction, of which one is an example of the method of invention, and which are described in the text which follows.

DETAILED DESCRIPTION OF THE INVENTION

The subject invention may be of utility in applications where extraction zone efficiency is hampered by extraction zone hydraulics, particularly in those applications where feed and/or recycle inlets are overloaded due to high hydrocarbon or solvent flow rates and/or deficiencies in the design of the extraction zone internals. This invention may be of particular utility for feeds having a relatively high non-aromatics content which are at least partially soluble in the solvent and which are difficult to remove in subsequent purification operations.

Referring to the Figure, an extractive separation process is shown. In the Figure all equipment, valves, piping and controls not necessary for an understanding of the present invention have been omitted for clarity. The internal construction of extractive separation zone 10 may be of conventional design, having trays, packed, baffled or mechanically agitated internals. A feedstream such as a hydrocarbon feedstock is shown passing through line 12 and thence through line 22 and nozzle 20, and/or through line 32 and nozzle 30 into extraction zone 10. Solvent is shown entering zone 10 through line 14. Raffinate comprising substantially no aromatics exits zone 10 through line 16 for further processing (not shown) while aromatics-rich extract containing some non-aromatic impurities exits through line 18. The extract passes into separation zone 40 having reboiler 50. The extract is separated into an overhead fraction and a bottoms fraction. The overhead fraction, comprising most of the non-aromatics, is condensed and recycled through line 42. The recycle then passes through line 24 and nozzle 20 and/or line 34 and nozzle 30 into extraction zone 10. Bottoms from separation zone 40 exit the base of zone 40 through line 44 for further separation of the solvent from the feed component in additional separation zones (not shown) before the solvent is returned to zone 10 through line 14. Part of the bottoms from line 44 are recycled through line 46 into reboiler 50 and thence through line 52 back to zone 40 to provide heat for the separation. The example of the method of the invention involves passing all the feed into the extraction zone 10 via line 32 and nozzle 30, and the introduction of some recycle via line 34 and nozzle 30, the remainder of the recycle being introduced into zone 10 via line 24 and nozzle 20. The other modes of operation are described for comparison only.

The subject invention has been used to improve the operation of an extraction zone used for the separation of benzene, toluene and xylene from non-aromatics using sulfolane as the solvent. In the application of this invention described hereinafter, zone 10 comprised a commercial extraction zone having perforated trays. Inlet lines 22 and 32 for feed, and 24 and 34 for recycle, permitted both the feed and/or recycle streams to be split before entering the extraction zone (for the purpose of demonstrating the benefits and advantages of the invention relative to non-inventive modes), rather than entering zone 10 through only one location. Nozzles 20 and 30 preferably are disposed a minimum of one tray apart. Preferably, at least 5 wt.% of the split stream is passed through each nozzle location. One measure of the efficiency of the extractive aromatics-non-aromatics separation process is the percent non-aromatics in a downstream BTX product line. Table I shows the net increase in the non-aromatics content of the downstream BTX product line over a base amount as a function of net increase in feed rate at constant recycle, where all the feed was added through line 32 and all the recycle was added through line 34 into nozzle 30 (not in accordance with the invention). Table II illustrates that the net increase in the non-aromatics content of the downstream BTX product line is much lower than the values reported in Table I when the feed rate is increased over a base amount and the feed is split, with a fraction of the feed being added through line 22 and a fraction through line 32, while all the recycle enters through line 34 (which operative mode is also not in accordance with the invention). From a comparison of Tables I and II, it can be seen that splitting the feed permits a greater increase in feed rate for substantially the same increase in the concentration of non-aromatics in the downstream BTX product line. In Table I, an increase in the feed rate of about 8.2% from the base rate resulted in an increase of 0.10 wt.% in the non-aromatics concentration in the downstream BTX product line. By comparison, when the feed was split, the data of Table II illustrate that an increase of about 16.4% from the base rate resulted in only a 0.11 wt.% non-aromatics content in the downstream BTX product line.

TABLE I

(NOT ILLUSTRATIVE OF INVENTION)
**INCREMENTAL CHANGE IN NON-AROMATICS
 CONTENT OF BTX PRODUCT WHEN ALL FEED
 ADDED THROUGH ONE FEED LOCATION**

	Feed Rate Increase to Extraction Zone Through Line 32 (wt.%)	Increase in Wt.% Non-Aromatics in Downstream BTX Product Line As Compared to Base Value
5	2.7	0
10	8.2	0.10
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TABLE II

(NOT ILLUSTRATIVE OF INVENTION)
**INCREMENTAL CHANGE IN NON-AROMATICS CONTENT OF BTX
 PRODUCT WHERE FEED ADDED THROUGH TWO INLET LOCATIONS**

	Wt.% Increase in Feed Rate Through Line 22 Over Base Rate in which All Feed Added Through Line 32	Increase in Wt.% Non-Aromatics in Downstream BTX Product Line as Compared to Base Value
20	0	0
25	2.7	0
30	3.7	0.03
35	6.4	0.04
40	9.1	0.04
45	12.8	0.05
	15.5	0.08
	16.4	0.11

As shown by the data in Table III, from another set of runs a much more dramatic increase in extraction zone performance could be realized by directing a fraction of the recycle through line 24 into zone 10 in accordance with the invention. The data indicate that where all the feed was added through line 32 and all the recycle through line 34 (not according to the invention), increasing the feed rate above the base rate resulted in a significant increase in the downstream non-aromatics content in the BTX product line. Increasing the feed rate to extraction zone 10 through line 32 by 12.3 and 21.5 wt.% resulted in increases of 0.04 and 0.14 wt.%, respectively, in the downstream non-aromatics content in the BTX product line. However, other data in this table demonstrated that splitting the recycle stream in accordance with the invention permitted a significant increase in the feed rate to zone 10 without causing a significantly adverse

effect on BTX product line quality. Where recycle stream 42 was split such that approximately 25 wt.% passed through line 24 and nozzle 20, and approximately 75 wt.% passed through line 34 and nozzle 30 while the feed rate was held constant through line 32 in accordance with one embodiment of the invention, the non-aromatics content of the BTX product line declined by 0.05 wt.%. With this same split of the recycle stream through lines 24 and 34 and with all the feed added through line 32, it was possible to increase the feed rate by more than 26 wt.% over the base rate while increasing the non-aromatics content of the BTX product line by only 0.02 wt.% over the base rate. Thus, it may be seen that splitting the recycle stream and adding a fraction of the recycle stream above the location at which all the feedstream is added permitted a very significant increase in product quality and/or in through-put.

10 In the extractive separation of benzene, toluene and xylene, the component most sensitive to the effectiveness of the extractive separation is xylene. Utilization of the above-noted process, in which a fraction of the recycle stream was split, produced about a 6% increase in xylene recovery as compared to a conventional process at comparable feed rates in which the recycle stream was not split. Thus, the addition of a fraction of the recycle above the location at which all the feed is added produced a downstream BTX product line having a reduced non-aromatics content while simultaneously increasing the xylene recovery as compared to a conventional process in which the recycle was not split.

15 While the examples have described a specific extraction system, it is clear that this invention is applicable to other extraction operations.

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TABLE III
INCREMENTAL CHANGE NON-AROMATICS CONTENT OF BTX
PRODUCT WHEN RECYCLE ADDED THROUGH TWO INLET LOCATIONS

Run No	Wt. % Increase in Feed Rate to Extraction Zone Over Base Rate; All Feed Added Through Line 32	Split of Recycle Stream to Extraction Zone (Wt. %)		Change in Wt. % Non-Aromatics in Downstream BTX Product Line
		Through Line 34	Through Line 24	
1	0	100	0	0
2	12.3	100	0	+0.04
3	21.5	100	0	+0.14
4*	0	75	25	-0.05
5*	1.5	75	25	-0.06
6*	26.2	75	25	+0.02

* Runs 4, 5 and 6 are illustrative of the invention.
 Runs 1, 2 and 3 are for purposes of comparison only, and are not illustrative of the invention.

Claims

1. A method for the extractive separation of an inlet feed stream comprising first and second components into raffinate and extract streams, comprising:
 - (a) introducing (32) the inlet feed stream (12) into an extraction zone (10);



TABLE III
INCREMENTAL CHANGE NON-AROMATICS CONTENT OF BTX
PRODUCT WHEN RECYCLE ADDED THROUGH TWO INLET LOCATIONS

Run No	Wt.% Increase in Feed Rate to Extraction Zone Over Base Rate; All Feed Added Through Line 32	Split of Recycle Stream to Extraction Zone (wt.%) Through Line 34 Through Line 24	Change in Wt.% Non- Aromatics in Downstream BTX Product Line
1	0	100	0
2	12.3	100	+0.04
3	21.5	100	+0.14
4*	0	75	-0.05
5*	1.5	75	-0.06
6*	26.2	75	+0.02

* Runs 4, 5 and 6 are illustrative of the invention.
 Runs 1, 2 and 3 are for purposes of comparison
 only, and are not illustrative of the invention.

Claims

1. A method for the extractive separation of an inlet feed stream comprising first and second components into raffinate and extract streams, comprising:
 - (a) Introducing (32) the inlet feed stream (12) into an extraction zone (10);



- (b) introducing (14) a solvent selectively miscible with one of the components into the extraction zone at a spaced-apart location from the location (30) at which the feed stream is introduced;
- (c) causing the feed stream and solvent stream to pass substantially countercurrently through the extraction zone (10) to produce an extract product stream (18) relatively rich in the first component and a raffinate product stream (16) relatively rich in the second component; and
- (d) returning a separate recycle stream (42), produced by subsequent processing (40) of at least one of the extraction zone product streams, into the extraction zone (10); characterized by:
- (e) introducing (24) a portion of the recycle stream (42) containing substantially all of the second component of the extract product stream into the extraction zone at a location (20) between the locations at which the solvent (14) and all the feed stream (30) are introduced into the extraction zone (10).
2. The method of claim 1 wherein the first component is an aromatic component and the second component is a non-aromatic component.
 3. The method of claim 2 wherein the solvent is selectively miscible with the aromatic component.
 4. The method of claim 2 or claim 3 wherein the solvent is selected from the group consisting of sulfolane, tetraethylene glycol, phenol, dimethyl-sulfoxide, furfural, N-methyl pyrrolidone and mixtures thereof.
 5. The method of any one of claims 1 to 4 wherein the solvent is introduced into the extraction zone at a location above the location at which the feed is introduced into the extraction zone.
 6. The method of any one of claims 1 to 5 wherein at least 5 wt.% of the recycled stream is introduced into the extraction zone at a location above the location at which all the feed is introduced into the extraction zone.
 7. The method of any one of claims 1 to 6 wherein the extraction zone comprises a trayed extraction zone, and wherein a portion of the recycle stream is introduced a minimum of one tray above the location where all the feed stream is introduced into the extraction zone.
 8. The method of any one of claims 1 to 6 wherein the extraction zone comprises a baffled extraction zone.
 9. The method of any one of claims 1 to 6 wherein the extraction zone comprises a packed extraction zone.

Revendications

1. Procédé de préparation extractive d'un écoulement d'alimentation d'entrée, comprenant des premier et second constituants, pour obtenir des écoulements d'un produit raffiné et d'un produit extrait, comprenant:
 - (a) l'introduction (32) de l'écoulement d'alimentation d'entrée (12) dans une zone d'extraction (10);
 - (b) l'introduction (14) d'un solvant miscible de façon sélective avec l'un des composants dans la zone d'extraction, en un emplacement distant de l'emplacement (30) auquel est introduit l'écoulement d'alimentation.
 - (c) la commande de l'écoulement d'alimentation et de l'écoulement du solvant pour qu'il traverse sensiblement à contre-courant la zone d'extraction (10) pour produire un écoulement (18) du produit extrait, relativement riche du premier constituant, et un écoulement (16) du produit raffiné, relativement riche, du second constituant, et
 - (d) le renvoi d'un écoulement séparé de recyclage (42) produit par un traitement ultérieur (40) d'au moins l'un des écoulements de produit de la zone d'extraction (10) dans cette zone; caractérisé par
 - (e) l'introduction (24) d'une partie de l'écoulement de recyclage (42) contenant sensiblement la totalité du second constituant de l'écoulement du produit extrait, dans la zone d'extraction en un emplacement (20) situé entre les emplacements, auxquels le solvant (14) et la totalité de l'écoulement d'alimentation (30) sont introduits dans la zone d'extraction (10).



2. Procédé selon la revendication 1, selon lequel le premier constituant est un constituant aromatique et le second constituant est un constituant non aromatique.
3. Procédé selon la revendication 2, selon lequel le solvant est miscible de façon sélective avec le constituant aromatique.
4. Procédé selon la revendication 2 ou 3, dans lequel le solvant est choisi dans le groupe comprenant le sulfolane, le tétraéthylèneglycol, le phénol, le diméthylsulfoxyde, le furfural, la N-méthylpyrrolidone et des mélanges de ces produits.
5. Procédé selon l'une quelconque des revendications 1 à 4, selon lequel le solvant est introduit dans la zone d'extraction en un emplacement situé au-dessus de l'emplacement au niveau duquel l'écoulement d'alimentation est introduit dans la zone d'extraction.
6. Procédé selon l'une quelconque des revendications 1 à 5, selon lequel au moins 5 % en poids de l'écoulement recyclé est introduit dans la zone d'extraction en un emplacement situé au-dessus de l'emplacement auquel la totalité de l'écoulement d'alimentation est introduit dans la zone d'extraction.
7. Procédé selon l'une quelconque des revendications 1 à 6, selon lequel la zone d'extraction comprend une zone d'extraction à plateaux, et selon lequel une partie de l'écoulement de recyclage est introduit à au moins un plateau au-dessus de l'emplacement où la totalité de l'écoulement d'alimentation est introduit dans la zone d'extraction.
8. Procédé selon l'une quelconque des revendications 1 à 6, selon lequel la zone d'extraction comprend une zone d'extraction à chicanes.
9. Procédé selon l'une quelconque des revendications 1 à 6, selon lequel la zone d'extraction comprend une zone d'extraction remplie d'un garnissage.

30 Patentansprüche

1. Verfahren zur extraktiven Trennung eines Einlaßmaterialstroms, der erste und zweite Komponenten umfaßt, in Raffinat- und Extraktströme, bei dem:
 - (a) der Einlaßmaterialstrom (12) in eine Extraktionszone (10) eingeführt wird (32),
 - (b) ein selektiv mit einer der Komponenten mischbares Lösungsmittel in die Extraktionszone an einer von der Stelle (30), an der der Einsatzmaterialstrom eingeführt wird, beabstandeten Stelle eingeführt wird (14),
 - (c) der Einsatzmaterialstrom und der Lösungsmittelstrom dazu gebracht werden, im wesentlichen im Gegenstrom durch die Extraktionszone (10) zu strömen, um einen Extraktproduktstrom (18), der relativ reich an der ersten Komponente ist, und einen Raffinatproduktstrom (16), der relativ reich an der zweiten Komponente ist, zu bilden, und
 - (d) ein separater Rückführungsstrom (42), der durch anschließendes Weiterverarbeiten (40) mindestens eines der Extraktionszonenproduktströme hergestellt wird, in die Extraktionszone (10) zurückgeführt wird,

dadurch gekennzeichnet, daß

 - (e) ein Teil des Rückführungsstroms (42), der im wesentlichen die gesamte zweite Komponente des Extraktproduktstroms enthält, in die Extraktionszone an einer Stelle (20) eingeführt wird (24), die zwischen den Stellen liegt, an denen das Lösungsmittel (14) und der gesamte Einsatzmaterialstrom (30) in die Extraktionszone (10) eingeführt werden.
2. Verfahren nach Anspruch 1, bei dem die erste Komponente eine aromatische Komponente ist und die zweite Komponente eine nicht-aromatische Komponente ist.
3. Verfahren nach Anspruch 2, bei dem das Lösungsmittel selektiv mit der aromatischen Komponente mischbar ist.



4. Verfahren nach Anspruch 2 oder Anspruch 3, bei dem das Lösungsmittel ausgewählt ist aus der Gruppe bestehend aus Sulfolan, Tetraethylenglykol, Phenol, Dimethylsulfoxid, Furfural, N-Methylpyrrolidon und Mischungen derselben.
5. Verfahren nach einem der Ansprüche 1 bis 4, bei dem das Lösungsmittel an einer Stelle in die Extraktionszone eingeführt wird, die oberhalb der Stelle liegt, an der das Einsatzmaterial in die Extraktionszone eingeführt wird.
6. Verfahren nach einem der Ansprüche 1 bis 5, bei dem mindestens 5 Gew.% des zurückgeführten Stroms an einer Stelle in die Extraktionszone eingeführt werden, die oberhalb der Stelle liegt, an dem das ganze Einsatzmaterial in die Extraktionszone eingeführt wird.
7. Verfahren nach einem der Ansprüche 1 bis 6, bei dem die Extraktionszone eine Böden enthaltende Extraktionszone umfaßt und ein Teil des Rückführungsstroms mindestens einen Boden oberhalb der Stelle eingeführt wird, an der der gesamte Einsatzmaterialstrom in die Extraktionszone eingeführt wird.
8. Verfahren nach einem der Ansprüche 1 bis 6, bei dem die Extraktionszone eine mit Einbauten versehene Extraktionszone umfaßt.
9. Verfahren nach einem der Ansprüche 1 bis 6, bei dem die Extraktionszone eine gepackte Extraktionszone umfaßt.