ABSORPTION AND EXTRACTION

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Commercial Extraction Equipment

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This paper presents an attempt by the authors to summarize all the available contemporary data on commercial extraction equipment. Their sources were the general literature, patent literature, and data from manufacturers and operators of extraction equipment. Several patented designs of such equipment have been reproduced herein.

THE literature on liquid-liquid extraction deals mainly with phase relationships, stage calculations, and discussion of the mechanism of mass transfer between two liquid phases. The published information on commercial extraction equipment, particularly the types used and performance data, is rather limited.

Hunter and Nash (*24*) have written several papers containing brief notes on extraction equipment. Two reviews by Waeser (*45*, *46*) written in Germany during the war have become available in this country. Green (*19*) in England reported on solvent extraction with special reference to fine chemical manufacture. However, the best discussions to date have been those of Elgin in Perry's Handbook (*38*) and his several annual reviews (*10*, *11*) published since that time.

This paper is an attempt to summarize all of the information available to date on commercial extraction equipment. The sources have been the general literature, patent literature, and data from manufacturers and operators of extraction equipment. The review of the patent literature is presented because it is a tedious process for an individual engineer and not very informative unless the reviewer goes back to the original patents. The patent literature contains little performance data on extractors, but does furnish many ideas on how extractors may be built and where they are operated. Only a few of the extractors described in the patent literature can be discussed in the paper. However, a complete listing of the patents reviewed and classified is given in the patent bibliography.

USE OF COMMERCIAL EXTRACTORS

The term "commercial extractor" in these discussions means a continuous countercurrent liquid-liquid contact device which is an essential part of a process operating to produce a chemical to supply a more or less steady demand. In Table I are listed the major processes in which the use of commercial extraction equipment has been reported. Most of these processes are well established in American industry, and the processes could not be operated at a comparable scale or as efficiently without satisfactory extraction equipment.

GENERAL CONSIDERATIONS FOR DESIGN OF EXTRACTION PROCESSES

As discussed in detail, and particularly as to theory, by Benedict (*1*), all physical separation processes essentially are ones in which power is expended to make a separation. An extraction process is only one of the several available means by which the power may be expended with reasonable efficiency for the desired purpose. Here the power expended is largely the heat required to recover the solvent. The primary limitations on the choice of a solvent are that its density be different from that of the diluent, that it be at least partially immiscible with the diluent, and that the solvent be recoverable. These basic limitations usually are less severe than those for an azeotropic agent, or those for a solvent for extractive distillation. Economic considerations add restrictions as to dissolving power of the solvent for the solute, selectivity, boiling point, chemical stability, and ease of separation from both the solvent and the diluent.

The cost of operating the extractor itself, which includes capital charges, labor, repairs, and power to extractor unit only, are but a small part of total cost of operating an extraction process. Major cost items in their approximate order of magnitude are:

1. The power (heat) required to separate the solvent from the extract and raffinate streams and to fortify it to a specified purity.

Table I. Major Commercial Applications of Liquid-Liquid Extraction Processes

I. Petroleum Refining
- A. Separating high purity products
  - Toluene
  - Butadiene
  - Olefins
- B. Separating one or more groups of compounds from cuts of wide boiling range
  1. Treatment of kerosene, Edeleanu (*93*)
  2. Lubricating oils treated with selected solvents
     - Edeleanu (liquid SO_2)
     - Chlorex [bis(2-chloroethyl)ether]
     - Duo-Sol
     - Furfural
     - Nitrobenzene
     - Phenol
- C. Dewaxing, deasphalting, and decarbonizing operations

II. Extraction of Acetic Acid
- A. Wood distillation
- B. Recovery of acetic acid from dilute solutions from cellulose acetate, etc.

III. Phenol Recovery
- A. Raschig process, primary phenol production
- B. Gas works liquids, recovery and by-product phenols
- C. Recovery of phenol from a wide variety of solutions

IV. Chemical Processes with Liquid-Liquid Contacting
- A. Nitration } Here the value of countercurrent operations may be
- B. Sulfonation } questioned because of the danger of over-reaction

V. Vitamins and Antibiotics

VI. Vegetable Oil Refining

VII. Fischer-Tropsch Synthesis of Liquid Fuels
- A. Separation of water-soluble by-products
- B. Separation of oil-soluble oxygenated by-products

2. Solvent loss. (This varies considerably.)
3. Capital and labor charges of the distillation equipment.
4. Capital, labor, and power for extraction equipment.

Since the processes usually are designed for minimum total over-all cost, the process design frequently is based on considerations which are not necessarily concerned with the extractor. This is particularly true where the purpose of the whole process is to obtain one or more materials of high purity, and the extraction step serves to remove interfering impurities to a concentration sufficiently low to permit the final purification by distillation.

Because of the wide choice of solvents possible for a given separation, one usually can find a solvent to give good selectivity; therefore, the extraction can be accomplished with a small number of theoretical contacts. Few commercial extractors have more than 10 equivalent contacts.

The above discussion should not be construed to mean that there is nothing to be gained by proper design and selection of extraction equipment. It merely suggests that the cost of operation of the various possible types of extractors will be close, and considerations not directly concerned with the extractor frequently dictate their final selection. The writers and the operators believe the major consideration is the selection of a design which gives the greatest freedom from operating and maintenance troubles. As an example, if there is a tendency for solids to form in the extractor, a simple mixer and settler extractor probably would be more desirable than a packed tower, unless the solid accumulation could be easily washed or dissolved from the packing.

Almost all of the existing successful commercial extractors operate year after year with total on-stream time of 95 to 99% of the elapsed time.

TYPES OF EXTRACTION EQUIPMENT

The scheme of Table II is suggested for classifying liquid-liquid extraction equipment. It is based on the methods used for bringing together and separating the phases and is subclassified according to type of contact. All known types of extractors can be readily classified by this scheme with but few examples where cross or double listing would be desirable.

The major classification is determined by whether or not gravity or centrifugal force is used to separate the phases. Most commercial extractors are of the gravity type.

Gravity separated extractors may be subclassified as to whether the contact between phases is through extended films or droplets. Here confusion may arise because in many cases contacting is done both through extended films and droplets.

The subclassification of the gravity-type extractor (group 1) as to form of interface is not so clear. In general, contacting is done with one phase continuous and the other spread out to give the necessary large interfacial area. This is done either by forming a film on a solid of suitable design, or by dispersing the discontinuous phase into droplets. In many designs of baffle tower, large surfaces are provided upon which the dispersed phase spreads as a film, and provision is made to direct the flow over the baffles so that there is appreciable contact through films. However, when the dispersed phase passes the edge of the baffle, it is broken into droplets. Sherwood, Evans, and Longcor (*42*) have shown in tne case of spray towers that an appreciable percentage of the total extraction occurs in the actual formation of droplets. Thus in columns in which both films and droplets are present, it is difficult or impossible to evaluate how much mass transfer is provided by the film and how much by the droplet. For this reason baffled towers, except those where special precautions are taken to prevent drop formation, have been considered droplet extractors.

Packed towers may operate with the discontinuous phase either spread out as a film over the packing or largely dispersed into droplets. Here the character of the packing surface, particularly as to wetting effect, surface tension, and viscosity of the two liquids, and their flow rates, all play an important part in determining the type of contact. In general, large packing, selectivity wetted by the discontinuous phase, together with high viscosity of the discontinuous phase and low flow rates, seem to favor film-type transfer. However, in glass laboratory extractors the operation of a packed column usually is accompanied by bubbles. Commercial-sized packed columns almost always employ nozzles or drilled distributors for the initial distribution of each phase and droplets are present. For this reason packed towers are considered as droplet extractors.

Table II. Classification of Extraction Equipment

(Liquid-liquid mass transfer equipment)

I. Two Phases Separated by Gravity		
A. Contact made through one phase as a film	B. Contact made with one phase dispersed as droplets	
	B_1. No additional power used to maintain dispersion or redispersion	B_2. Extra power used to redisperse or maintain dispersion
1. Extended surfaces	1. Spray tower	1. Mixer and settler
2. Packed towers Large packing grids	2. Baffle tower 3. Perforated plate 4. Bubble cap 5. Packed tower	2. Baffle tower with movable members
II. Two Phases Separated by Centrifugal Force		

Droplet extractors may be divided into two types, those which require no power except that available in the streams of liquid going to the extractor, and those which use additional outside power to maintain the dispersion, or to redisperse the dispersed phase. The largest number of designs and patents for extractors are for those which do not use outside power. This type of extractor has several advantages over other types. In addition to saving power necessary for redispersion, there is no maintenance of stuffing boxes, long shafts, etc. The apparent trend has been toward this type, and it is safe to predict the trend will continue, perhaps at an accelerated rate, when reliable performance data become more widely distributed and accepted. When additional

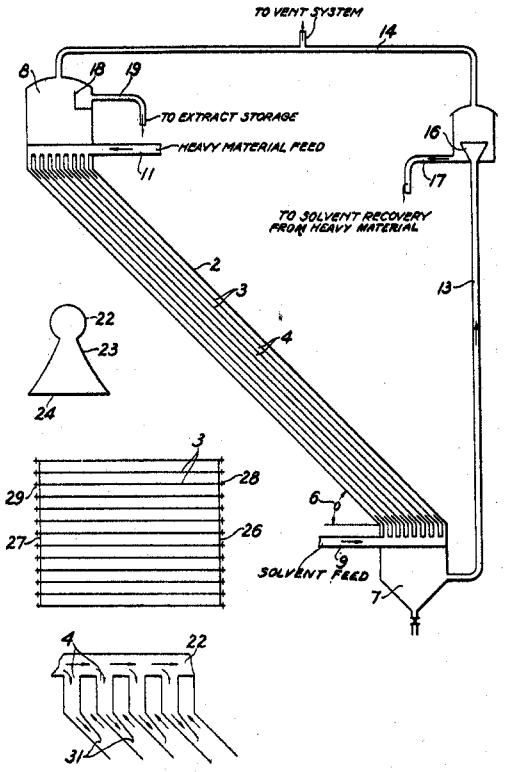

Figure 1. Film-Type Extractor (49)

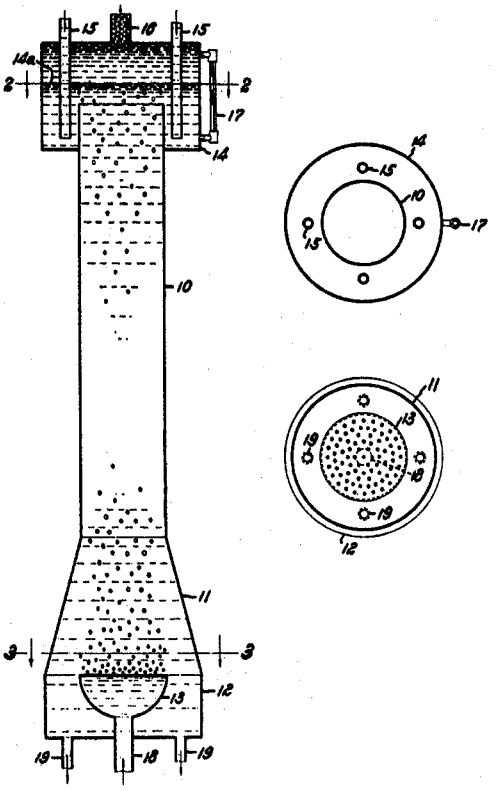

Figure 2. Spray Tower with Tapered End (50)

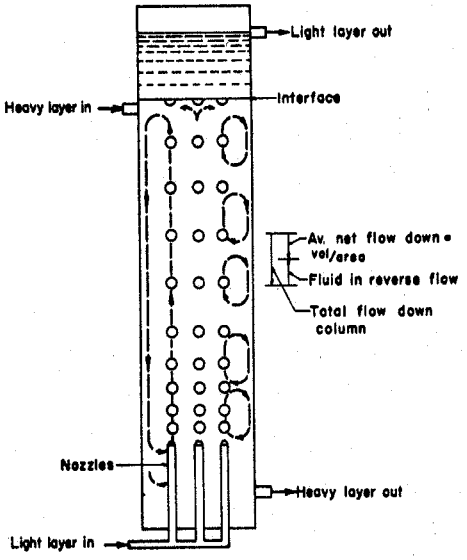

Figure 3. Simplified Drawing of Spray Column

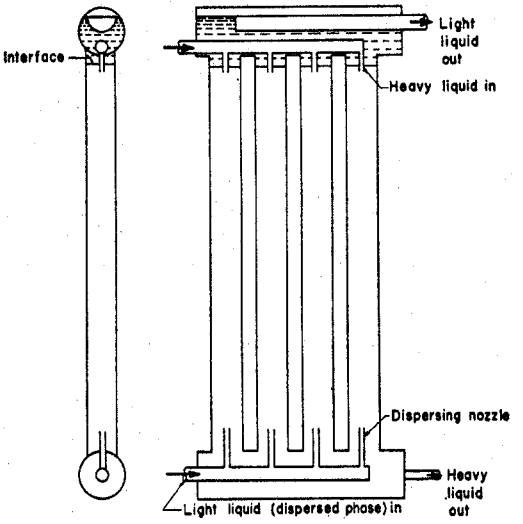

Figure 4. Spray Tower Extractor
Extractor of Table III

power is supplied to redisperse or maintain dispersion, the extractor is essentially a mixer and settler extractor.

DESIGN AND PERFORMANCE OF COMMERCIAL EXTRACTORS

The performance of 47 extractors, classified according to the scheme of Table II, is given in Table III. The data for nine of these come from Dow operations or from data collected by Dow engineers.

In Table III an attempt has been made to give the important details of design: the system on which the column operates; throughput expressed as cubic feet of liquid of each phase per hour per square foot of horizontal empty cross section (which is equivalent to the average linear velocity in feet per hour); and the efficiency, expressed as number of defined unit sections of column per theoretical stage contact. Average phase densities at the operating temperatures are given. Since these densities will

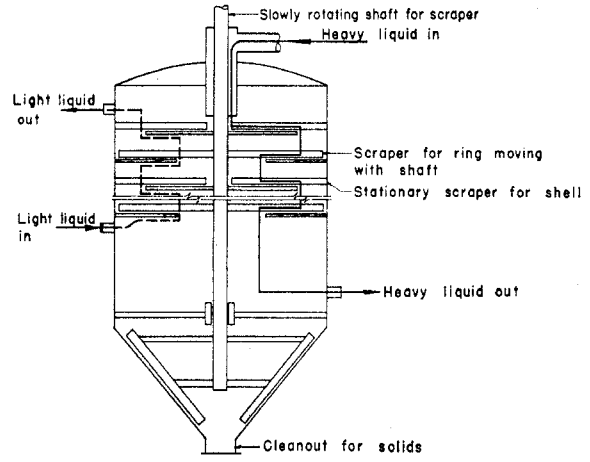

Figure 5. Baffle Tower with Scrapers

vary within the column owing to variations in temperature and solute removed and distribution of solvent and diluent between the two phases, averages of the densities at the top and bottom of the unit have been taken. The vertical flow velocities also vary considerably within the unit due to partial miscibility of the two phases. The flows reported are the maximum flows for each phase to be found in the extractors. In the case of mixer-settler extractors, the flows in the settling chamber have been calculated as though they occurred in the vertical direction.

The efficiencies so reported should be regarded as approximations. Many of the extractors were originally designed to give a definite number of theoretical contacts for a specified product. Most extractors reported have delivered the specified product, although sometimes operating at different conditions from design, and the performance in terms of theoretical contacts has never been determined. In asking for performance data the authors found many people reluctant to furnish analyses for concentration changes or equilibrium data, primarily because they did not consider them accurate enough for publication. If the product meets specifications, which can be determined by some relatively simple test, not necessarily translatable into numbers of analytical significance, the operator considers the extractor to be operating satisfactorily.

DISCUSSION OF SPECIFIC TYPES OF EXTRACTORS

Film-Type Extractors. One gravity extractor was found that could be classed as a true extruded surface film-type of extractor This has been patented by Gordon and Zeigler (*49*). The inventors (*17*) report that it was never built on a commercial scale.

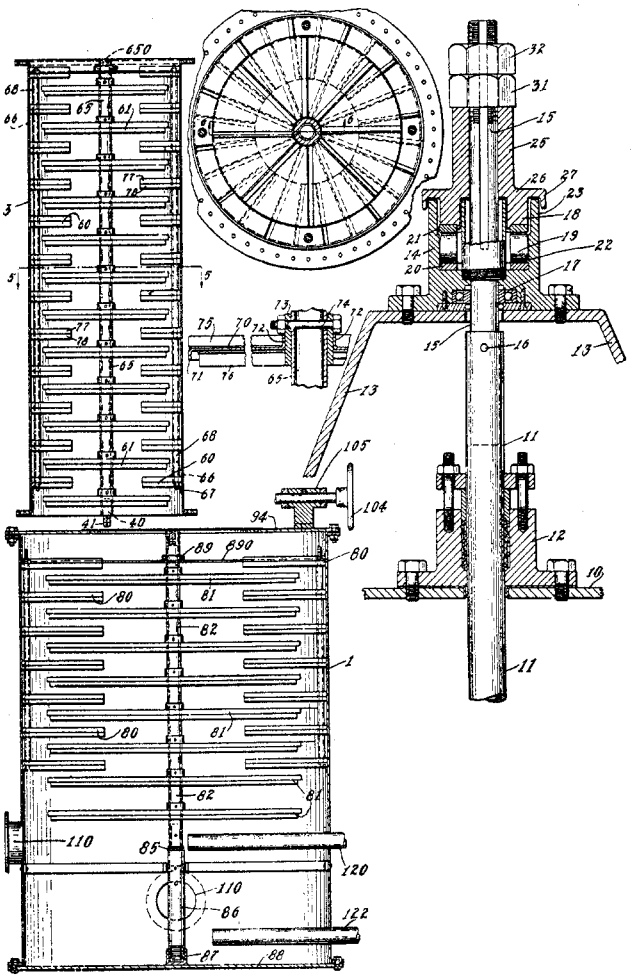

Figure 6. Coahran (53) Modification of Donut and Disk-Type Baffle Tower

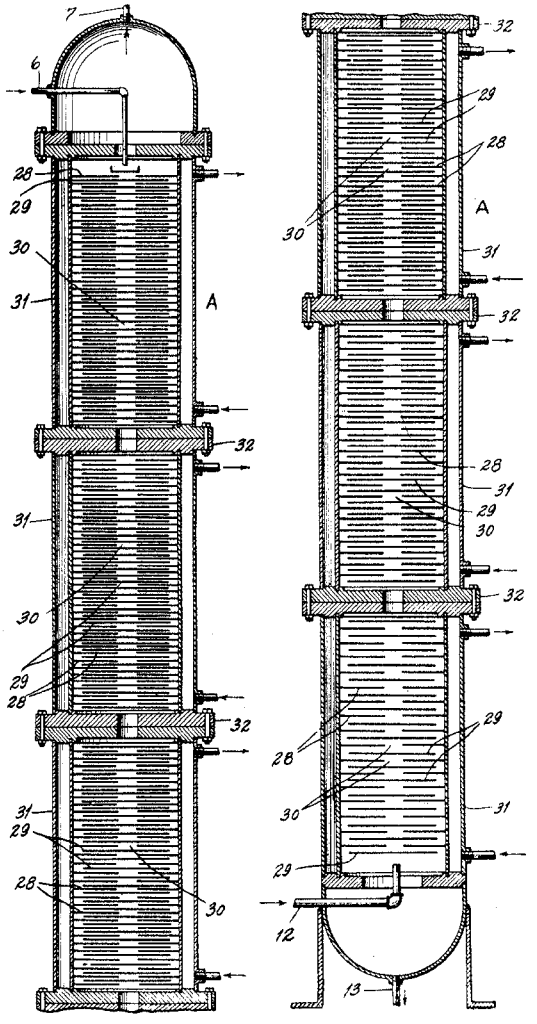

Figure 7. Dons *et al.* (54) Modification of Donut and Disk-Type Baffle Tower

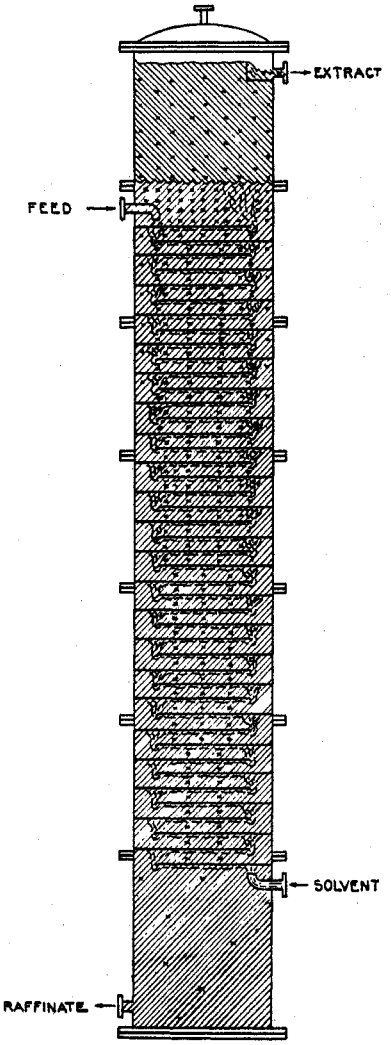

Figure 8. Baffle Plate Extractor

However, data of the experimental unit is listed as extractor 1 in Table III because of the high efficiency reported. This apparatus, shown in Figure 1, is built up from elongated cells or ducts which are very broad and long relative to their thickness. The ducts are inclined at an angle to induce layer formation within each individual duct. The main advantage claimed for this design is that it will handle the two phases with a minimum of emulsification. However, the throughput rates are lower than those obtainable in a comparable vertical column.

Spray Towers. The spray tower is perhaps the cheapest and simplest extractor. Although it has been extensively studied on a laboratory scale, as far as the writers can learn it has not been widely used commercially. Many of the spray-type columns used commercially employ redispersion and are really a type of mixer and settler column. One of the few commercial applications is that used in the Columbia alkali process (*44*) for extracting sodium chloride and sodium chlorate to make "rayon grade" caustic soda from electrolytic soda. Liquid ammonia is the solvent. This is extractor 2 in Table III.

From the authors' experience, based primarily on observations in glass columns 4 inches in diameter and short glass sections of decanters 12 inches in diameter, there are two problems presented in the operation of spray columns. These are:

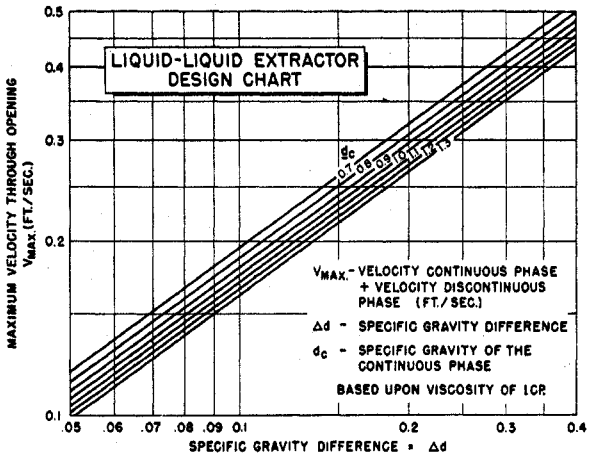

Figure 9

1. The tendency of the droplets of the dispersed phase to coalesce.
2. Recirculation in the continuous phase.

Elgin (*50*) has patented a design with a tapered end (Figure 2) for the incoming dispersed phase which reduces the effect of coalescence. However, the inventor (*12*) has stated that his design has not been used to date for any large or commercial extractor.

Recirculation in the continuous phase may be caused by thermal currents, density differences, or simply by the friction of the moving bubbles carrying some of the continuous phase along with them. In this case the average flow of the continuous phase, set by its rate of feed, is the difference between a forward velocity greater than average, and the backward flow caused by any of the above-mentioned causes. This means some of the liquid moves in the direction of flow at a rate greater than the average, and there is some by-passing. These are shown schematically in Figure 3. One would expect this effect to increase with column diameter. One solution to the problem is to build the extractor as a number of small diameter columns in parallel, as shown in Figure 4. Extractor 3, of Table III, is such a unit. Extractor 34, of Table III, was built to operate as a spray tower but did not give satisfactory performance without the packing.

Baffle Towers. Baffle tower extractors are simply vertical towers provided with baffles to direct the liquid flows over baffle surfaces and then over the baffle edge to the next set of baffles. A common type is the "donut and disk"-type where one baffle,

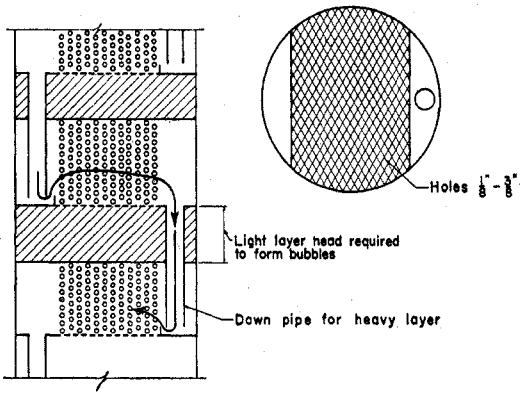

Figure 10. Perforated Plate Column
Perforations in horizontal plates

Table III. Commercial

Item[a]	Service	Description of Extractor	Solvent	Diluent or Feed	Solute	Average Densities: Heavy layer	Average Densities: Light layer	Average Densities: Difference	Temp., ° F.: Top	Temp., ° F.: Bottom	Size of Extractor: Section, diam. in ft.
											GRAVITY SEPARATED,
											(Contact in Commercial Centrifugal Ex
1	Acetic acid recovery from wood distillation	1/4 × 1 inch cross section, 15 ft. length, 45° angle	n-propyl acetate / n-propyl alcohol	Wood liquor	Acetic acid	...	...	...	...	...	...
											GRAVITY SEPARATED,
2	NaOH purification	Spray column	Liquid NH_3	50% aqueous NaOH	NaCl and $NaClO_3$	1.53	0.52	1.01	160	160	3
3	Phenol recovery	Spray column, 8-inch pipes in parallel	Benzene	Water	Phenol	1.01	0.89	1.12	125	125	0.7
											GRAVITY SEPARATED
4	Acetic acid recovery from wood distillation	Donut and disk, slow moving scrapers for solids	Ethyl and methyl acetate	Wood dist. liquor	Acetic acid	1.01	0.88	0.13	110–120	110–120	3.5
5	Acetic acid recovery in cellulose acetate plant	Donut and disk, 3.5 ft. i.d., 1.5 ft. hole	Ethyl acetate	Water	Acetic acid	1.01	0.88	0.13	100–130	100–130	3.5
6	Hydrocarbon oil	Center to side baffle tower	...	...	...	...	...	0.20	...	...	6.5
7	Hydrocarbon oil	Center to side baffle tower	...	...	...	...	...	0.21	...	...	3.5
8	Hydrocarbon oil	Side to side baffle tower	...	...	...	...	...	0.10	...	...	2.2
9	Propane deasphalting	Parallel grid, baffle tower	Propane	Reduced crude oil	Nonasphaltenes	1.00	0.70	0.30	160	110	10
10	Propane deasphalting	Parallel grid, baffle tower	Propane	Reduced crude oil	Nonasphaltenes	1.00	0.70	0.30	155	117	10
11	Propane deasphalting	Parallel grid, baffle tower	Propane	Reduced crude oil	Nonasphaltenes	1.00	0.70	0.30	157	121	11
											GRAVITY SEPARATED
12	Hydrocarbon oil	(Perforated plates with multiple redispersion on each plate)	Furfural	Hydrocarbon oil	Asphaltenes	0.97	0.85–0.90	0.07–0.12	...	...	...
13	Hydrocarbon oil		Furfural	Hydrocarbon oil	Asphaltenes	0.97	0.85–0.90	0.07–0.12	...	...	...
14A	Phenol–lube oil	19-tray perforated plate tower	Phenol	Neutral distillate oil	Naphthenes	...	...	...	160	110	8
14B	Phenol–lube oil	19-tray perforated plate tower	Phenol	150 viscosity residuum oil	Ashpaltenes and naphthenes	...	...	...	230	155	8
15	Phenol–lube oil	20-tray perforated plate tower	Phenol	Deasphalted oil	Asphaltenes and naphthenes	1.05	0.90	0.15	195	173	12
16	Phenol–lube oil	20-tray perforated plate tower	Phenol	Deasphalted residuum oil	Asphaltenes and naphthenes	1.03	0.88	0.15	240	200	9
17A	Phenol–lube oil	14-tray perforated plate tower	Phenol	Neutral distillate oil	Naphthenes	1.06	0.91	0.15	170	160	12
17B	Phenol–lube oil	14-tray perforated plate tower	Phenol	Dewaxed distillate oil	Naphthenes and aromatics	1.05	0.89	0.16	175	165	12
18	Phenol–lube oil	.....	Phenol	Neutral distillate oil	Naphthenes	1.05	0.90	0.13	175	147	11
19	Phenol–lube oil	.....	Phenol	Neutral distillate oil	Naphthenes	1.04	0.89	0.15	190	161	11
20	Phenol–lube oil	20-tray perforated plate tower with downcomers and uptakes	97% phenol	Neutral distillate oil	Naphthenes	1.00	0.88	0.12	170	145	10
21	Phenol–lube oil	...	Phenol	Distillate oil	Asphaltenes and naphthenes	1.03	0.84	0.19	155	115	8
22	Phenol–lube oil	...	Phenol	Distillate oil	Asphaltenes and naphthenes	1.01	0.83	0.18	180	160	13
23	Phenol–lube oil	...	Phenol	Distillate oil	Asphaltenes and naphthenes	1.00	0.83	0.17	255	210	12
24	Propane deasphalting	16-tray perforated plate tower	Propane	East Texas residuum oil	Nonasphaltenes	0.95	0.70	0.25	160	120	10
25	Butane washing	30-tray perforated plate tower	Water	Aqueous acetone Butane	Acetone	1.00	0.70	0.30	...	...	5

[a] A and B numbers refer to same extractor under different operating conditions.

(*This section continued on page 1028*)

Extraction Equipment

Size of Extractor: Total height, ft.	Size of Extractor: Unit defined	Size of Extractor: No. of units	Total No. of Theoretical Contacts	Unit/Theoretical Stage Contact	Vertical Flow Cu. Ft./(Hr.)(Sq. Ft.): Light layer	Vertical Flow Cu. Ft./(Hr.)(Sq. Ft.): Heavy layer	Volume Ratio light/heavy	Reflux ratio, Reflux/Feed	Concentration Changes	Equilibrium Data	Continuous Phase	Remarks	References
Contact through Films													
tractors Is Mainly through Films)													
...	1-ft. length	15	~20	~0.75	...	...	...	...	...	...	...	Only gravity film-type extractor reported, never built in commercial size	(17, 49)
Droplet Contact without Power, Spray Column													
40	1-ft. tower	40	...	...	32.5	37.1	0.88	None	2.0% NaCl → 0.2% 0.2% $NaClO_3$ → 0.0002%	...	Upper, liquid NH_3 Lower, NaOH	...	(44)
20	1-ft. tower	20	~7	~3	...	...	0.56	None	8% phenol → 0.4%	$x = 0.01$, $y = 0.022$ $x = 0.02$, $y = 0.08$	Aqueous	...	(8)
Droplet Contact without Power, Baffle Tower													
45	1 donut + 1 disk	43/tower	6	10.75	60–75	30	2.0–2.5	None	8.75% AcOH → 0.05%	$x = 0.0405$, $y = 0.0875$ $x = 0.0004$, $y = 0.0048$	Aqueous	Performance for two towers in series	(5, 16)
...	1 donut + 1 disk	65	~8	~8	65–75	25	2.5–3.0	None	22% AcOH → 0.11 AcOH	...	Aqueous	...	(31)
48	1 tray	103	~12	~8	Total flow both phases	64.9	...	None	...	...	...	5-inch tray spacing	(47)
48	1 tray	104	~12	~8		81.2	...	None	...	...	...	5-inch tray spacing	(47)
36	1 tray	96	~12	~8		92.1	...	None	...	...	...	4-inch tray spacing	(47)
53	1-ft. grid ht.	...	Probably 2–3	~20	78.5	4.2	17.7	None	...	...	Propane	Grids similar to open floor grating. Extraction need not be very efficient	(22)
41	1-ft. grid ht.	...	Probably 2–3	~20	63.3	2.7	23.6	None	...	...	Propane		(22)
41	1-ft. grid ht.	...	...	~20	64	3.9	17.0	None	...	...	Propane		(22)
Droplet Contact without Power, Perforated Plate Tower													
...	1 tray	6	1.2–2.8	5.0–2.1	44.5	66.6	0.67	...	...	...	..	16-inch tray spacing	(23, 66)
...	1 tray	...	...	1.8	238	...	...	...	...	...	...	Carryover was 0.069% by volume of light phase	(23, 66)
61	1 tray	19	...	...	6.5	8.2	0.79	0.7	...	...	Upper, raffinate Lower, extract	...	(18, 27)
61	1 tray	19	...	...	5.2	9.9	0.53	0.7	...	...	Upper, raffinate Lower, extract	...	(18, 27)
60	1 tray	20	...	...	7.2	26.8	0.26	...	...	...	Upper, raffinate Lower, extract	...	(29)
53	1 tray	20	...	...	6.9	52.5	0.13	...	...	...	Upper, raffinate Lower, extract	Perforated shower deck baffles	(15)
47	1 tray	14	4–6	3.5–2.3	16.3	79.5	0.21	...	...	...	Upper, raffinate Lower, extract	Feed introduced on 4th plate	(50, 62)
47	1 tray	14	4–6	3.5–2.3	15.4	88.5	0.17	...	...	...	Upper, raffinate Lower, extract	Feed introduced on 4th plate	(50, 62)
65	1 tray	...	2	...	11.1	25.9	0.43	...	...	...	Upper, raffinate Lower, extract	...	(22)
47	1 tray	...	1.4	...	8.1	25.1	0.32	...	...	...	Upper, raffinate Lower, extract	...	(22)
60	1 tray	20	...	...	13.1	18.9	0.69	None	...	...	Upper, raffinate Lower, extract	Downcomers in oil phase packed to coalesce entrained extract	(20)
37	1 tray	...	5	...	11.3	14.4	0.79	...	...	...	Upper, raffinate Lower, extract	...	(55)
58	1 tray	...	15	...	16.6	30.3	0.55	...	...	...	Upper, raffinate Lower, extract	Special step-type trays	(35)
58	1 tray	...	15	...	9.1	19.3	0.47	...	...	...	Upper, raffinate Lower, extract	Special step-type trays	(35)
39	1 tray	16	...	...	108.1	9.6	11.3	None	...	...	..	Perforated shower deck baffles over entire area	(15)
80	1 tray	30	...	...	38.5	36.2	1.06	None	10% acetone → 0.01%	...	...	2.5-ft. plate spacing	(65)

(This section continued on page 1028)

Table III. Commercial

Item[a]	Service	Description of Extractor	Solvent	Diluent or Feed	Solute	Average Densities: Heavy layer	Average Densities: Light layer	Average Densities: Difference	Temp., ° F.: Top	Temp., ° F.: Bottom	Size of Extractor: Section, diam. in ft.
											GRAVITY SEPARATED,
											(No Known Com
											GRAVITY SEPARATED,
26	Ammonia from butane	1-inch Raschig rings	Water	Butylene	Ammonia	0.92	0.62	0.30	120	80	1
27	Vegetable oil refining	1/2-inch ceramic Berl saddles	Furfural	Degummed soybean oil	Unsaturated glyceride oils	1.16	0.95	0.21	80	80	1.8
28	Vegetable oil refining	1/2-inch ceramic Raschig rings	Furfural	Refined linseed oil	Unsaturated glyceride oils	1.14	0.96	0.18	80	80	5.6
29	Furfural-lube oil	...	Furfural	400 viscosity Texas distillate oil	Asphaltenes	1.10	0.89	0.21	229	196	13
30	Furfural-lube oil	1 1/4-inch ceramic Raschig rings	Furfural	Distillate oil	Asphaltenes	1.10	0.86	0.24	260	170	5.6
31A	Furfural-lube oil	1-inch Raschig rings	Furfural	Dewaxed neutral oil	Naphthenes	1.10	0.87	0.23	230	160	...
31B	Furfural-lube oil	1-inch Raschig rings	Furfural	Dewaxed bright stock oil	Naphthenes	1.10	0.90	0.20	290	225	...
32	H_2S from propane	1 × 1 1/4 inch, 20 B.W.G. steel Raschig rings	Diethanolamine	Propane	H_2S	1.08	0.47	0.61	115	110	
33	H_2S from propane	1 × 1 1/4 inch, 20 B.W.G. steel Raschig rings	Diethanolamine	Propane	H_2S	1.08	0.47	0.61	115	117	3.6
34	Cyanohydrin	1/4-inch carbon Raschig rings	Methyl ethyl ketone	Brine liquor	Ethylene cyanohydrin	1.20	0.86	0.34	60–75	60–75	1.7
36	Phenol recovery	5/8-inch ceramic Raschig rings, 2 towers in series	Benzene	Water	Phenol	1.01	0.89	0.12	125	125	3.3
36	Phenol recovery	5/8-inch ceramic Raschig rings	Benzene	Water	Phenol	1.01	0.89	0.12	125	125	6.7
37	Phenol recovery	1-inch ceramic Raschig rings, 2 brick-lined towers	Benzene	15% HCl	Phenol	1.07	0.89	0.18	140	140	1.3
38	Edeleanu-kerosene	1-inch Raschig rings	Liquid SO_2	Kerosene	Aromatics	1.38	0.89	0.49	10	−5	4.6
											GRAVITY SEPARATED,
39	Phenol recovery	2 × 2 ft. mixer; total agitator power, 2.3 hp.	Chlorobenzene	Water	Phenol	1.00	1.10	0.10	110	110	Settler, 2 × 10 ft.
40	Phenol recovery	8 × 8 in. mixer; total agitator power, 1.5 hp.	Chlorobenzene	Water	Phenol	1.00	1.10	0.10	110	110	Settler, 6 × 1 ft.
41	Phenol recovery	4.25 × 8 ft. mixer; 130 r.p.m. agitators	Light creosote oil	Gas liquor	Monohydric phenols	1.02	0.95	0.07	66–77	66–77	Settler, 8.5 ft. diam.
42	Phenol recovery	4.25 × 8 ft. mixer; 130 r.p.m. agitators	5% NaOH solution	Creosote oil extract	Monohydric phenols	1.10	0.95	0.15	66–77	66–77	Settler, 9 ft. diam.
43	Duo-Sol-lube oil	Mixer, horizontal settler	Phenol-cresol and propane	Mixed reduced crude oils	Paraffins and aromatics	1.05	0.80	0.25	120	100	8 ft. diam., 2 at
44	Duo-Sol-lube oil	Mixer, horizontal settler	Phenol-cresol and propane	95 V.I. bright stock oil	Paraffins and aromatics	1.05	0.80	0.25	130	120	...
45	Nitrobenzene-lube oil	Mixer, horizontal settler	Nitrobenzene	Lube distillate oil	Naphthenes	1.10	0.90	0.22	55	50	7.75
46	Nitrobenzene-lube oil	Mixer, horizontal settler	Nitrobenzene–H_2SO_4	Dewaxed oil	Naphthenes	1.20	0.90	0.30	60	45	5
47	Butadiene–butylenes	Mixer, horizontal settler with 3 horizontal baffles	Ammoniacal copper acetate	Butylenes–butane	Butadiene	1.10–1.20	0.61	0.49–0.59	25	25	Settler, 6.5 ft. diam.

[a] A and B numbers refer to same extractor under different operating conditions.

the "donut," is a ring with a large diameter hole in the center which makes tight contact with the shell. Above and below, or on each side of the ring, there is a flat disk, usually somewhat larger than the hole in the ring. This type has been used for many years in extracting acetic acid from the aqueous demethanolized liquors from wood distillation. In the design shown in Figure 5, the "disks" are rotated at low speed against fixed scrapers, and the shaft that carries the disk also carries scrapers to scrape the stationary "donut." These scrapers move any solids that form in the extractor to the edge of the baffle and eventually to the bottom of the tower where provisions are made to remove them. Extractor 4 in Table III is of this design.

Although the original installations of this type of extractor were made before 1930, some recent patents have been obtained on modifications. In a design by Coahran (*53*) the "donut" sections are stationary, while the "disk" sections may be rotated to provide some agitation (Figure 6). In another design (Figure 7) by Dons, Mauro, and Mapes (*54*) the distance between the "donut" and "disk" plates is progressively decreased toward the upper portion of the column to decrease in turn the thickness of the layers as they pass from relatively naphthenic phases (in the specific case of solvent extraction of a petroleum oil stock with Chlorex) in the lower part of the column to more paraffinic phases in the upper portion of the column. The variations in thickness of the different layers change the ratio of the extended film surface to volume of the two phases and is claimed to result in greater

Extraction Equipment (*Continued*)

Size of Extractor: Total height, ft.	Size of Extractor: Unit defined	Size of Extractor: No. of units	Total No. of Theoretical Contacts	Unit/Theoretical Stage Contact	Vertical Flow Cu. Ft./(Hr.)(Sq. Ft.): Light layer	Vertical Flow Cu. Ft./(Hr.)(Sq. Ft.): Heavy layer	Volume ratio light/heavy	Reflux Ratio, Reflux/Feed	Concentration Changes	Equilibrium Data	Continuous Phase	Remarks	References
DROPLET CONTACT WITHOUT POWER, BUBBLE CAP TOWER													
mercial Applications to Date)													
DROPLET CONTACT WITHOUT POWER, PACKED TOWER													
20	1-ft. packing	15		~7.5	19.0	4.0	4.7	None	10% NH_3 → 0.3%	...	Aqueous	Shell is water cooled for heat of reaction of $NH_3 + H_2O \rightarrow NH_4OH$	(8, 59)
87	1-ft. packing	75	...	...	2.5	20.3	0.12	2	...	...	Upper, oil Lower, furfural	...	(14, 88)
67	1-ft. packing	...	...	...	2.8	20.4	0.14	0.6	...	...	Upper, oil Lower, furfural	...	(14, 88)
47	1-ft. packing	...	...	...	4.9	17.9	0.28	...	...	...	Oil	6 redistribution sections	(29)
63	1-ft. packing	40	...	...	13.2	66.0	0.20	0.35	...	...	Oil	4–10 ft. packed sections with 4 redistribution sections	(6)
...	1-ft. packing	60	...	...	26.2	61.4	0.43	0.35	...	...	Oil	10–5 ft. packed sections with 10 redistribution sections	(8, 7)
...	1-ft. packing	60	...	...	15.6	59.0	0.27	0.50	...	...	Oil	10–5 ft. packed sections with 10 redistribution sections	(8, 7)
50	1-ft. packing	34	...	...	58.2	26.7	2.2	...	3–5 mole% H_2S → 0%	...	Propane	Feed is 60 vol. %; C_3, 40 vol. % C_4	(30)
48	1-ft. packing	30	...	...	92.5	42.5	2.2	...	2 mole% H_2S → 0%	...	Propane	Feed is 15 vol. %; C_3, 85 vol. % C_4	(30)
34	1-ft. packing	26	4.5	5.8	16.6	14.7	1.13	None	Brine 17.5% → 2.0% Methyl ethyl ketone 0% → 19.0%	...	Brine	90% removal of cyanohydrin from brine soln.	(8)
30	1-ft. packing	23/tower	4.5+2.5	5.1 in rich tower, 9.2 in lean tower	11.8	20.8	0.56	None	3.5% phenol → 0.2%	$x = 0.01$ $y = 0.022$ $z = 0.02$ $y = 0.08$	Aqueous	...	(8)
59	1-ft. packing	53	7	7.5	11.8	20.8	0.56	None	3.5% phenol → 0.2%	...	Aqueous	...	(8)
35	1-ft. packing	30/tower	2 + 1.3	15 in rich tower, 23 in rich tower	7.8	13	0.6	None	0.4% phenol → 0.09%	$y/x = 8$	Aqueous	Each tower supplied with fresh benzol	(8)
22	1-ft. packing	14.5	...	...	17.2	111	1.55	None	17.8 vol.% aromatics → 7.2%	...	Kerosene	...	(30)
DROPLET CONTACT WITH POWER REQUIRED FOR REDISPERSION, MIXER-SETTLER													
4	Mixer-settler	8	6	1.33	8.0	to 8.0	0.3–1.0	None	5.0% phenol → 0.1%	...	...	...	(8)
3	Mixer-settler	8	6	1.33	7.2	5.0	1.43	None	8.0% phenol → 0.1%	...	...	...	(8)
18 ft. length	Mixer-settler	5	...	...	13.1	13.1	1.0	...	2.9% phenol → 0.2%	$y/x = 4–5$	...	...	(33, 43, 86)
18 ft. length	Mixer-settler	4	...	...	12.4	12.4	1.0	...	...	...	...	...	(33, 43, 86)
75 ft. length	Mixer-settler	8	...	...	...	...	2.07	...	...	...	...	Propane and cresol flow countercurrent to each other through series of stages, feed near center	(5, 6)
...	Mixer-settler	7	...	...	...	...	...	...	...	...	...		
33 ft. length	Mixer-settler	6	...	...	6.4	10.5	0.61	...	...	...	...	...	(15)
10	Mixer-settler	4	...	...	9.85	10.6	0.93	...	...	...	...	Mixed liquid divided into 3 streams in settler	(88)
30 ft. length	Mixer-settler	7–8	5–8	1.4–1.0	0.35–0.9 in each section	7 in each of 4 sections	0.05–0.24	...	20 to 99*% and 20 to 1	...	...	...	(55)

selectivity of treat. A simple design (*47*) of baffle column which is commercially available employs side-to-side flow, or center-to-side flow in large columns, of the liquid phases, comparable to that of the liquid in a conventional bubble cap distillation column. This is shown in Figure 8. Extractors 6, 7, and 8 in Table III are of this design. Although the contact efficiency is low, of the order of 0.05 to 0.10 theoretical contact per baffle, the baffles can be closely spaced 4 to 6 inches apart and reasonable contact efficiency for the whole tower is obtained.

The capacity of a given baffle column is limited by the flooding which occurs at the opening in the baffles through which the two phases must pass. An empirical design chart given by Wentworth (*47*) plots a maximum allowable velocity through the horizontal passage between baffles, expressed as the sum of the velocities of the two phases, against the difference in density of the phases, with the density of the continuous phase as a parameter (Figure 9). This chart applies specifically to liquids with viscosities of the order of 1 centipoise and with interfacial tension of the magnitude of that of benzene-water. Similar charts for liquids of other viscosities may be readily prepared from data obtained in laboratory equipment. The capacity of this design of baffle tower thus is seen to increase with greater difference in density between the two phases and with decreasing density of the continuous phase.

In many respects the baffle column is one of the most satisfactory types of commercial extractors. There are no small holes or

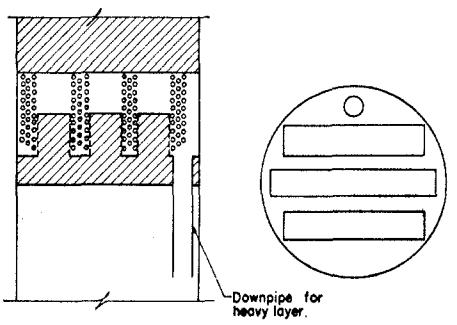

Figure 11. Perforated Plate Column
Perforations in vertical plates

passages to plug or to be enlarged by corrosion. If the liquids used are clean and form no precipitates, this type of extractor operates with very low maintenance. However, if there is any accumulation of solids that cannot be removed by solvent, and unless special provisions for clean-out openings are made, the cost of dismantling for cleaning makes them impractical.

Perforated Plate Columns. These extractors also are quite simple. In this type the dispersed phase is redistributed into droplets many times by passage through plates perforated with small holes. The dispersed phase accumulates, either above or below the perforated plate, until sufficient hydrostatic head is generated to force the liquid through the perforations, which break the stream into droplets. The droplets pass through the continuous phase until they are caught by the next plate and the process repeated. Figure 10 shows a column design with the perforations in the horizontal plane (*65*).

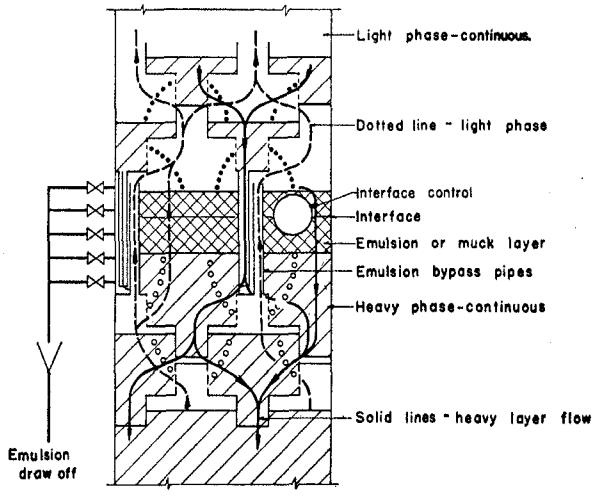

Figure 12. Emulsion By-Pass

The type of perforated plate shown in Figure 11 is one of the most widely used columns of this type. The perforated plate in this design is in the vertical plane, so that there is some sacrifice of height per tray. However, with holes in the vertical plates, the holes are to some extent protected from solids, which can move along with the continuous phase. Columns 14 to 20 of Table III are of this design. The perforated plate column frequently is operated with the interface level near the middle of the column, so that each phase in its particular part of the column is the dispersed phase.

When solids are carried into or form in an extractor, they usually accumulate at the interface to form almost a third phase of a muck or emulsion layer. When this occurs, provision can frequently be made to remove this continuously or periodically while the extractor is in operation. This emulsion drawoff is shown in Figure 12. Here the continuous phase is carried into a chamber in which the interface is maintained, with by-pass pipes for each phase, and with suitable side draw connections. This scheme cuts into the operation of the two adjacent trays (because there is some by-passing). The muck layer is removed from the extractor and the solids removed by breaking the emulsion in any of a number of ways, and filtering out the solids.

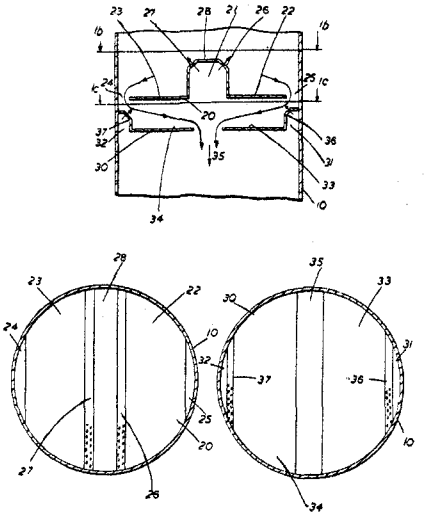

Figure 13. Modification of Perforated Plate Design
Trough-type design (*68*)

There are many modifications of perforated plate designs. Figure 13 shows a trough-type design patented by the Standard Oil Development Company (*68*). This is extractor 25 in the table. It is claimed that recycling is reduced in this design. One of the most recent innovations in perforated plate-type extractors is the design shown in Figure 14 (*66*). It was originally developed as a distillation contact plate, but has been found suitable for liquid-liquid extraction. The dispersed phase travels horizontally

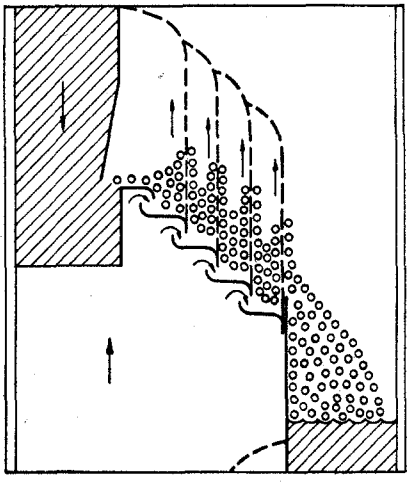

Figure 14. Modified Perforated Extraction Tray

into a high velocity stream of the continuous phase. The droplets are swept up against and through a perforated vertical baffle and are redispersed in a second stream of the continuous phase. The process is repeated several times across the plate. Up to 0.75 theoretical contact per tray is claimed with high throughput. Extractors 12 and 13 in Table III are of this type.

Perforated plate columns, particularly those with the perforations in vertical plates, have been quite satisfactory where they have been installed. They are very popular in the oil industry.

Bubble Cap Columns. Rogers and Thiele (*40*) report that bubble cap columns are not very efficient contact devices for liquid-liquid extraction. In spite of this, a number of patents have been granted on this type of contactor (*69–73*). The caps or liquid distributors disclosed in these patents often are of very unique design. Bubble cap columns probably can be built at a cost equivalent to that of baffle or perforated plate columns, if one recognizes the fact that 5 to 10 plates may be required to obtain a theoretical contact, and the plates can be spaced at close intervals.

The writers have found no commercial applications of bubble cap columns for extractors. A number of reports of such installations were traced, but invariably they have been found to be perforated plate towers.

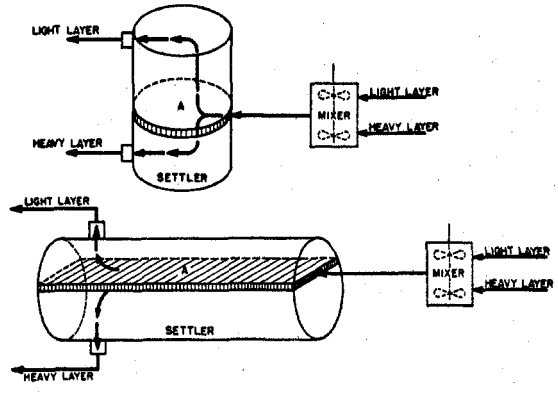

Figure 15. Flows in Mixer-Settler Extractors

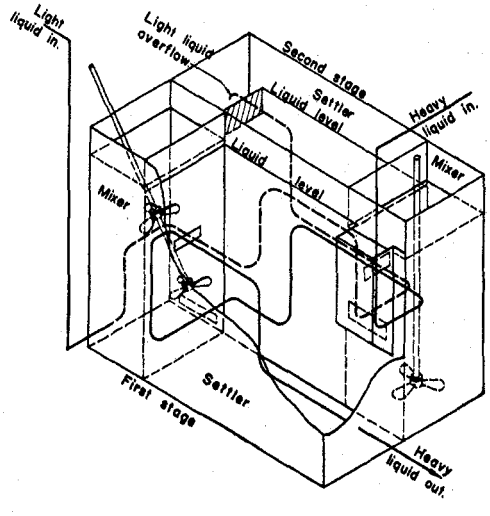

Figure 16. Box-Type Mixer-Settler Extractor

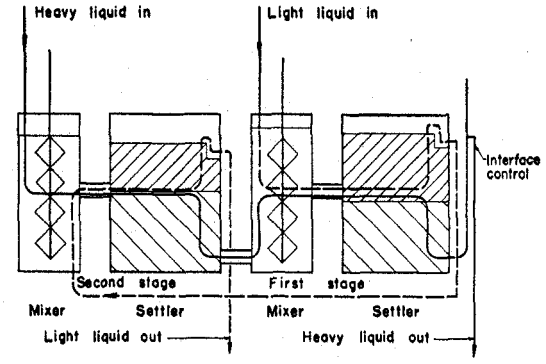

Figure 17. Two-Stage Mixer-Settler Extractor

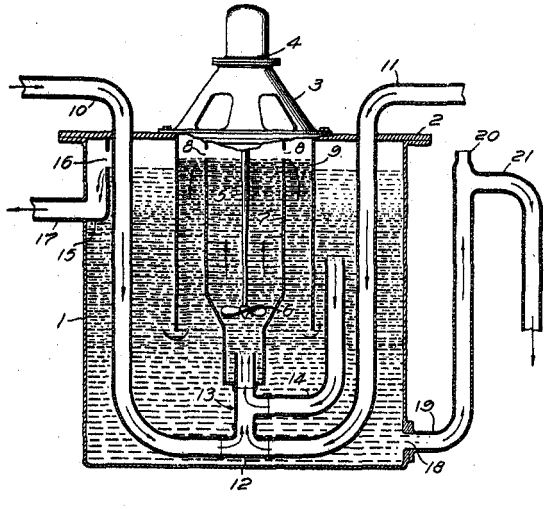

Figure 18. Mensing (108) Design for Mixer-Settler Extractor

Packed Tower Extractors. Packed towers rank next to spray towers in simplicity of construction. They are simply vertical tanks provided with a grid to support the packing and distributors for the two liquids. The favorite packings are Raschig rings and simple grids. The packing cuts down recirculation of the continuous phase, gives redispersion of droplets, and forms extended films. It is not difficult to determine their capacity as far as flow of the two liquids is concerned, but it is difficult to predict their efficiency, or feet of packing required per theoretical contact, except on the basis of the performance of a rather large pilot unit. As shown in Table III, this figure ranges for 5 to 20 feet. Extractors 26 to 38 in the table are packed tower installations.

In the cases where their performance has been demonstrated, they have been rather satisfactory, and many duplicate or even larger units have been built. In the petroleum industry they are the favored type for extraction with sulfur dioxide and furfural. Edeleanu plants have gone from mixer and settlers to packed towers. They also have been used successfully for phenol extraction, particularly in the Raschig phenol process.

The packed tower, built with a brick-lined tower and using porcelain, stoneware, or carbon packing, is probably the cheapest and most satisfactory extractor to use on very acid solutions. In packed towers, although the height per contact seems high, the equilibrium conditions are frequently very favorable and good extraction is obtained.

Mixer-Settler Extractors. Extractors in this class consist of a mixing chamber of suitable design followed by a settling chamber

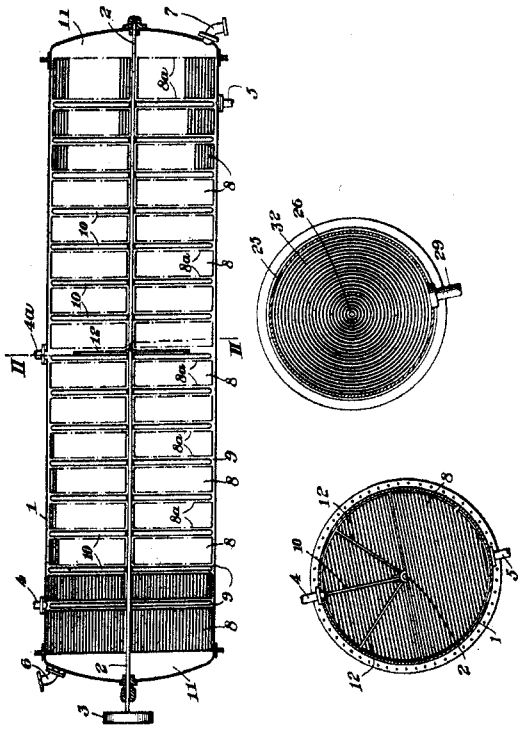

Figure 19. Bottaro (89) Design for Mixer-Settler Extractor

for each stage of the extractor. The mixer sometimes is only a centrifugal pump, but usually is a chamber provided with a propeller or turbine agitator and baffles. The settling chamber is simply a tank, although it is sometimes provided with elaborate baffles, or filled with packing. Figure 15 shows a typical stage

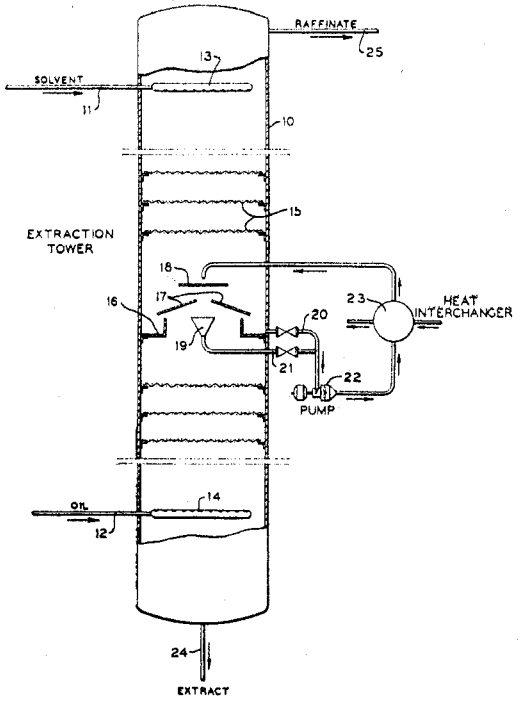

Figure 20. Kiersted (101) Design for Mixer-Settler Extractor

unit, in which the settling chamber can be a vertical or horizontal cylindrical tank. The larger extractors are built with horizontal tanks. The stages may be mounted side by side or one above the other. Extractors 39 to 47 in the table are mixer-settler units in commercial use.

In almost all cases where data is obtainable, contact efficiencies of the order of 0.75 to 0.9 contact per theoretical stage are reported, regardless of the size of extractor or the liquids upon which they operate. For this reason a conservative assumption of stage efficiency can be made. Thus the mixer-settler extractor may be considered in extraction to be analogous to the bubble cap column or distillation. It is the safest type to use in designing extractors from laboratory data. Almost the only design problem is to provide ample settling chambers.

For many pairs of liquids there is an easily discernible settling velocity, below which clear streams can be obtained. However, each stream will carry the other phase in amounts in excess of the solubility. It is carried as small droplets or "haze" which will separate in time, but so slowly that the design based on this rate would be economical. Usually mixer settlers operate with the hazy phases. However, if such a hazy solution passes through a bed where it contacts fine filaments such as glass wool, woven wire mesh, screens, etc., coalescence occurs and the excess second phase drops out. Burtis and Kirkbride (4) gave a good example of the use of fiber glass to coalesce dispersions of petroleum and water. The use of fibers, packing, or baffles in settlers to reduce by-passing and promote coalescence is quite common.

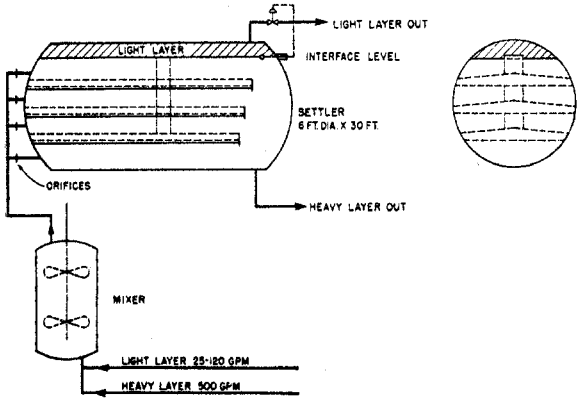

Figure 21. Butadiene Extractor

One design for a mixer-settler extractor is that shown in Figures 16 and 17 as a two-stage unit. The origin of the design is lost, although the authors have tried to trace it. The earliest record that can be found is its use in 1904 by E. O. Barstow, of The Dow Chemical Company, to extract benzoic acid from an aqueous solution with toluene. The first unit was made of wood. This extractor is readily adapted for construction from wood, metals, or from brick, so that it can be used for almost any pair of liquids. A rectangular mixing chamber with propeller agitators on either a vertical or inclined shaft is provided at the end of a settling chamber of the same width. The units are mounted side by side, with the mixers at alternate ends. The light liquid on one side flows over a weir and is carried to the mixer. From the other side, at the bottom of the settling chamber, the heavy liquid enters the mixing chamber at the bottom. The mixed liquids flow through an opening in the partition between the mixer and the settler. This opening is baffled to reduce turbulence in the settling chamber.

The flow of liquid liquid from stage to stage is maintained by locating the light liquid level overflow weirs on each mixer lower

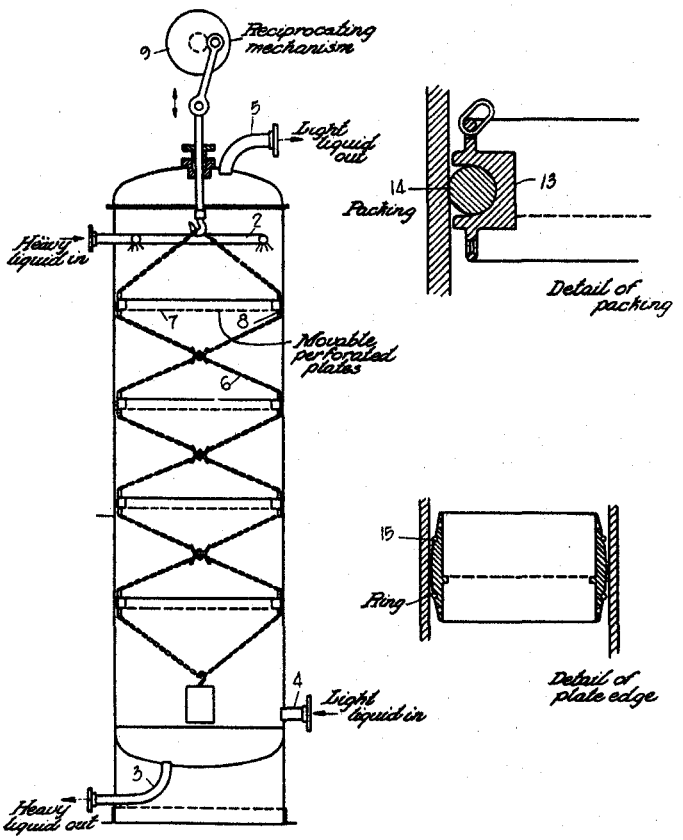

Figure 22. Van Dijck (120) Design for Extractor Column Movable Members

than on the preceding one. The flow of the heavy layer is maintained by a difference in level of interface from settler to settler as shown in Figure 17. This interface gradient establishes itself and varies with the flow rates. The only design precaution is to have sufficient depth of liquid in the first settler to allow a depth of light layer of about 1 foot in that stage. With a total depth of 42 inches of liquid, these extractors have been built in units up to 8 stages.

The design of Mensing (*108*), Figure 18, is similar except that the lift of liquid by the impeller transfers the mixture from stage to stage, and any number of units can be mounted at the same level. Several large units of this type have been built and have given very satisfactory performance.

When the stages are mounted one above the other, as in the case of one patented by McConnell (*103*), the mixer chambers can be built inside the extractor, and the power supplied to the agitators by a shaft running through the length of the tower. Othmer's (*109*) design is similar. The very efficient Scheibel extractor (*41*) is essentially this type with the added feature of using packing in the settling section.

The Bottaro (*89*) extractor (Figure 19) is a mixer-settler of unusual design, which is inclined from the vertical at an angle to obtain better layer separation. The settling section is heavily baffled.

Another scheme used is to make the tower simply a series of settlers one above the other and to remove the liquids from the tower at each level, mix them, and return them. This is shown in the Kiersted design (*101*) in Figure 20. In this design the return flow may be passed through heat exchangers to maintain desired temperature gradients. This is one way of providing reflux within the extractor.

Many of the largest extraction units in operation are mixer-settler units. The Duo-Sol process (*117*), which distributes the different components of a hydrocarbon feed between a phenolic solvent and propane, uses mixer and settler extractors (*5*). Extractors 43 and 44 of Table III are of this type.

A large fraction of the butadiene produced in this country is produced in the plants designed by the Standard Oil Development Company, in which butadiene is extracted from the other C_4 hydrocarbons by a solution of ammoniacal copper acetate. In this process the extraction system, patented by Packie and Glazier (*36*, *37*), and described by Asbury (*32*), utilizes an extractor of the type shown in Figure 21. Its performance data is given as extractor 47.

Perhaps the most important development in very large mixer-settler extractors is the use of interface level controllers to maintain the interface level. This permits positive movement and control of the flow of both phases, regardless of

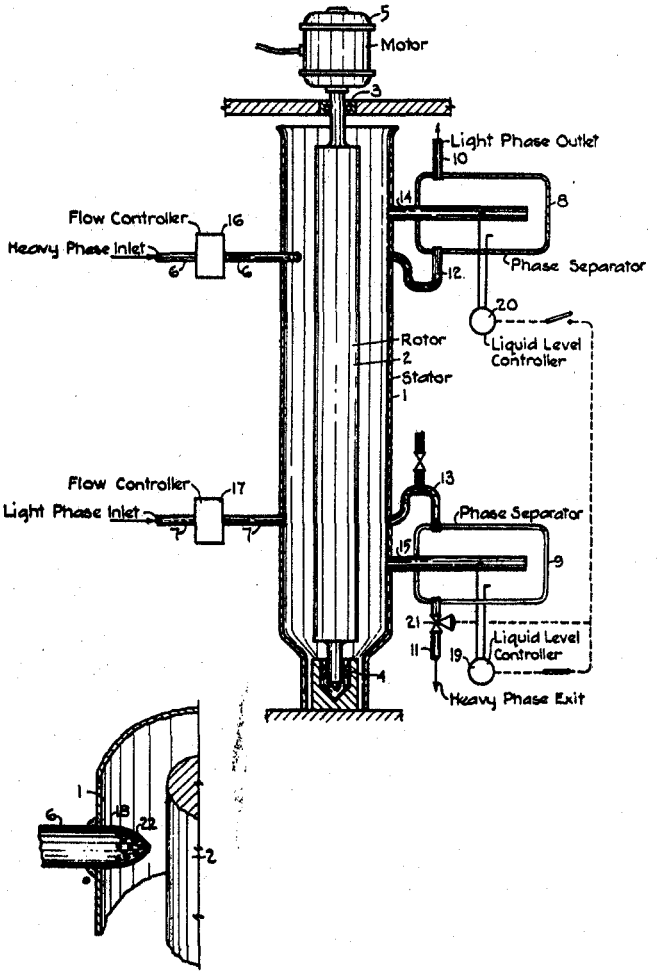

Figure 23. Maycock (107) Design for Extractor Column with Cylindrical Rotating Contact Surfaces

fluctuations in density of the phases. It also provides a constant inventory of each phase in the extractor, which allows smooth and even operation of the distillation equipment used with the extraction system.

Extraction Columns with Movable Members. A very unusual method of supplying power for redispersion is given in a patent by van Dijck (*120*). The design is shown in Figure 22. This is a perforated plate column in which the plates are raised and lowered by a reciprocating mechanism through the main body of liquid.

Extracting Columns with Cylindrical Rotating Contact Surfaces. Another unusual extractor recently patented by Maycock (*107*) utilizes the principle applied a few years ago by Rossini (*48*) in distillation, in which the annular space between a fixed wall and a rotating cylinder is used for contact. Figure 23 shows such a design.

Centrifugal Extractors. It is only in the last few years that extractors of the centrifugal or second group have been used. They must be carefully designed and constructed to withstand severe mechanical stresses and therefore are expensive. They have limited capacity and require careful maintenance. This type of extractor has one outstanding advantage over the gravity type. It can be built with a high throughput to inventory ratio. A unit with a 10,000-gallon-per-hour throughput for both streams can be built with an inventory of 10 gallons, which is equivalent in residence time in the extractor to 3.6 seconds. This feature is quite important in handling materials which are easily decomposed, or which undergo spontaneous degradation in the process liquors. Such conditions are found in the new antibiotics and vitamins. It is here that these machines are used to best advantage. The writers have been informed that today there are about 400 of these machines in operation, some installations consisting of banks of 10 machines. No attempt has been made by the present authors to cover or list the literature pertaining to centrifugal extractors.

OPERATION AND CONTROL OF EXTRACTORS

Whenever possible extractors are operated with the continuous phase either as the cheapest phase or the safest phase, even though this may require extra capital for the extractor—for example, if the inventory of a flammable solvent can be reduced to 20% of what it would otherwise be, it would be well worth extra expenditure for more contacts if this selection of the continuous phase gave lower efficiency.

Extractors are quite stable in operation, with the flow of the feed, solvent, and reflux set by flow controllers, and the solvent and reflux flows set at values adequate for the most adverse feed concentration conditions. It is quite fortunate that they are so stable, because the ratio of inventory to flow is usually high, and concentration changes follow changes in operating conditions with such a great time lag that it would be very difficult to attempt to make changes to effect control.

When the product goes below established standards, it does so slowly, and this means usually that the feed is definitely off or that the extractor needs cleaning or repairs. In plants which operate with regularly scheduled shut downs for repairs, and the cleanup and repairs are done carefully, failure of products of extractors to meet established standards is a rare event.

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