

Changes shown by ↕

CONTENTS

Section	Page
SCOPE	2
REFERENCES	2
INTERNATIONAL PRACTICE	2
OTHER REFERENCES	2
DEFINITIONS	2
APPLICATION	3
DESIGN CONSIDERATIONS	3
SELECTION OF DISPERSED PHASE	3
TYPES OF PACKING AND MATERIAL	4
PACKING SIZE	4
TOWER DIAMETER	4
TOWER HEIGHT	5
Height Equivalent of a Theoretical Stage (HETS)	5
Liquid Redistribution	5
Interface Detection and Control	5
TOWER INTERNALS	6
Packing Supports (Dispersers and Redistributors)	6
Bed Limiters	6
Inlet Distributors	6
Coalescence Media	7
DRAWING NOTES	7
DESIGN PROCEDURE	7
NOMENCLATURE	8
TABLES	
Table 1 Calculation Form (Customary Units)	9
Table 2 Calculation Form (Metric Units)	13
FIGURES	
Figure 1 Typical Packed Extraction Tower (Light Phase Dispersed)	17
Figure 2 Extractor Packing Flood Curve	18
Figure 3 Internals for Dispersed Phase (Light Phase Dispersed)	19
Figure 4 Internals for Continuous Phase Inlet (Light Phase Dispersed)	20

Revision Memo

12/99	Design criteria for interface control zone made consistent with those for perforated (sieve) tray towers, Section IV-B . Applications window has been better defined.
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DESIGN PRACTICES		EXTRACTION TOWERS PACKED EXTRACTION TOWERS PROPRIETARY INFORMATION - For Authorized Company Use Only	EXXON EXXON ENGINEERING
Section IV-C	Page 2 of 20		
Date December, 1999			

SCOPE

The procedure in this section covers the general techniques for specifying the diameter, height, and internals for packed liquid-liquid extraction towers.

- All equations in the text of this section are numbered in the same way as they appear on the Calculation Forms provided in **Table 1** or **Table 2** (Metric). Wherever possible, both the customary and the metric equations are shown in the text, the latter shown with an "M". However, if the equation is complex, the metric version has been omitted in the text for clarity but can be found on the Calculation Form (Metric).

REFERENCES

INTERNATIONAL PRACTICE

IP 5-2-1 Internals for Towers, Drums and Fixed Bed Reactors

► OTHER REFERENCES

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DEFINITIONS

Disperser	The hardware device that generates small droplets of dispersed phase which then enter the packing.
Emulsion Band	The emulsion band is the concentrated layer of dispersed phase droplets which accumulate at the interface between the continuous phase and the coalesced dispersed phase. The droplets accumulate at the interface until drainage of the film of continuous phase which surrounds the droplets is adequate to permit coalescence.
Flooding	Flooding is a condition caused by operation of the extraction tower at dispersed or continuous phase flow rates exceeding the capacity of the tower. Typically, flooding is evidenced by a significant buildup of the dispersed phase within the extractor, excessive entrainment, or unstable operation.
Phase Inversion	Phase inversion refers to a condition where the dispersed phase droplets spontaneously coalesce, trapping the continuous phase which originally surrounded the droplets. This causes the continuous phase to become dispersed as fine droplets in the coalesced dispersed phase. This phenomenon primarily occurs in emulsion bands which have a high concentration of dispersed phase droplets. It can also occur at high dispersed phase flow rates and cause flooding.
Solvent Treat Ratio	The solvent treat ratio is the ratio of the volumetric flow rate of solvent to that of the feed. It is generally expressed as a percentage, for example, 10% amine treat.

	EXTRACTION TOWERS PACKED EXTRACTION TOWERS		DESIGN PRACTICES	
	PROPRIETARY INFORMATION - For Authorized Company Use Only		Section IV-C	Page 3 of 20
			Date December, 1999	

APPLICATION

In general, packed towers should only be considered for extraction services requiring tower diameters less than 3 ft (1 m), where installation of perforated (sieve) trays are not practical or economic. Experience and owner preference may indicate packing usage for some applications with larger diameters. Exxon has commercially operated packed towers as large as 9 ft (3 m) in diameter. The service should be free of solids because fouling of the packing can be an issue. Materials selection is important since metallic packings typically have no tolerance for corrosion. In addition, turnaround procedures need to consider potential ignition of pyrophoric material (12). Since there is limited experience with packed extraction towers, it is recommended that new designs be checked by the Fractionation and Thermodynamics Section of ER&E.

Packed extractor designs described in this section are typically used to remove trace contaminants, such as hydrogen sulfide, from light hydrocarbon liquids, such as LPG. **Figure 1** shows a typical packed extractor design, with the light hydrocarbon feed introduced near the bottom by a distributor and dispersed by a support plate disperser. After flowing through the lower packed bed, the hydrocarbon is collected under the support plate/redistributor and redispersed before flowing up through the second packed bed. The heavier aqueous phase is the continuous phase and is introduced near the top of the extractor. The hydrocarbon and aqueous phase flow rates through the tower are essentially constant since very little mass transfer occurs. The liquid phases separate at the top of the tower where the interface is controlled. Emulsion-stabilizing contaminants, such as surface active chemicals and fine solids, can significantly broaden the emulsion band at this interface and cause upsets.

For new applications, slip-stream, pilot plant, or laboratory testing may be needed to establish the mass transfer and emulsion forming characteristics of the system. Special designs may be needed if a significant fraction of a feed is extracted, for example, greater than 10%. These systems can have low interfacial tension and, therefore, tend to form very small droplets and emulsions which are difficult to separate. In addition, flow rates inside the tower can vary considerably. It is recommended that the Fractionation and Thermodynamics Section of ER&E be consulted regarding these designs or the design of any extractor where the interfacial tension is below 10 dyne/cm (10 mN/m).

The table below, which is based on operating experience, lists the lower and upper operating limits for packed liquid-liquid extractors. If your case does not fall within these limits contact the Fractionation and Thermodynamics Section of ER&E.

PACKED LIQUID-LIQUID EXTRACTOR OPERATING LIMITS

VARIABLE	LOWER LIMIT	UPPER LIMIT
Pressure	40 psia (275 kPa)	300 psia (2100 kPa)
Temperature	100°F (40°C)	200°F (95°C)
Diameter	4 in. (0.1 m)	3 ft (1 m)
Physical Properties:		
• Interfacial tension	10 dyne/cm or (mN/m)	50 dyne/cm or (mN/m)
• Liquid viscosity	0.1 centipoise or (mPa • s)	2 centipoises or (mPa • s)
• Specific Gravity Differential	0.1	1
Bed Height	18 in. (0.45 m)	lower of 5 column diameters or 15 ft (4.6 m)
Packing Type	Pall rings, or demonstrated equivalent	
Packing Size	1 in. (25 mm)	Less than 1/8 column diameter and no more than 2 in. (50 mm)
Service (fouling tendency)	Clean service only	

DESIGN CONSIDERATIONS

SELECTION OF DISPERSED PHASE

The first consideration in designing an extraction tower is the selection of the phase to be dispersed. An important guideline is to disperse the phase that does not wet the packing and other internals, in order to minimize droplet coalescence and maximize efficiency. Metal internals are typically used by Exxon and these are usually wet by the aqueous phase. Another guideline is to disperse the phase with the higher volumetric flow rate since this will provide the largest number of drops and thus maximize the interfacial contact area between the two phases. For most Exxon applications, the hydrocarbon rate is much higher than the aqueous phase rate. Therefore, Exxon's experience with packed extractors is primarily with the hydrocarbon phase dispersed. One could consider dispersing the aqueous phase if it were extremely viscous or if one of the phases is at supercritical conditions. Consultation with the Fractionation & Thermodynamics Section of ER&E is recommended in such cases.

DESIGN PRACTICES		<i>EXTRACTION TOWERS</i> PACKED EXTRACTION TOWERS PROPRIETARY INFORMATION - For Authorized Company Use Only	 EXXON ENGINEERING
Section IV-C	Page 4 of 20		
Date December, 1999			

DESIGN CONSIDERATIONS (Cont)

TYPES OF PACKING AND MATERIAL

The same packings available for vapor-liquid service could, in principle, be used for liquid-liquid extraction; however, Exxon only has experience with random packings. Initially Raschig rings, Berl saddles and ceramic Intalox saddles were used as packings in extraction services. More modern random packings, such as Pall rings, have also been successfully applied. The relative capacities and efficiencies demonstrated by various random packings in distillation are not applicable to extraction. Screening studies have shown extractors with structured packing to be more expensive than those with random packing for grass-roots designs. Structured packings may offer some capacity and efficiency advantages for debottlenecks; however, commercial-scale data are sparse. The Fractionation and Thermodynamics Section of ER&E should be consulted for any application of structured packing in extractors.

Intalox saddles are preferred for ceramic packings and Pall rings for metal or plastic. Metal packings manufactured under the trade names of Flexi Rings and Ballast Rings (Koch-Glitsch) are considered equivalent to Pall Rings (Norton) for design purposes. Choice of packing material will be influenced by corrosion issues as well as solubility and wetting characteristics of the specific system. In order to maintain high interfacial area, a packing material should be selected that is not wetted by the dispersed phase. Metal packings are typically used where the hydrocarbon phase is dispersed. Ceramic packings are also not wetted by hydrocarbon, whereas plastic is wetted by hydrocarbon. Plastic packings are rarely used since high temperatures, especially during process upsets or steam-outs, and the presence of hydrocarbon can cause melting or dissolution.

Merichem fiber packing may also be a viable option in special situations, for example, when the interfacial tension is low or there is a tendency to form slow-breaking emulsions and only a few theoretical stages are needed. The Fractionation and Thermodynamics Section of ER&E should be consulted if novel packing types such as this are being considered for use.

PACKING SIZE

It has been observed (5) that for packings above a certain critical size, the droplet size passing through the packing is independent of the original droplet size formed by the disperser and virtually independent of the packing size and phase rates (up to the flooding point). Below this critical packing size, however, droplets tend to be temporarily trapped in the packing, producing unstable operation. It is therefore recommended that the packing size be larger than this critical size as calculated by [Eq. \(4b\)](#) or [\(4b\)M](#) in the Calculation Form. This equation predicts the critical packing size as a function of interfacial tension and density difference between the phases. There have been reports (3) that the critical packing size is sometimes larger than calculated in [Eq. \(4b\)](#) or [\(4b\)M](#). Therefore, packings smaller than 1 in. (25 mm) should be avoided if possible.

If interfacial tension information is not available, contact the Fractionation and Thermodynamics Section of ER&E where it can be estimated or measured. Measurement is preferred to estimation because interfacial tension is strongly affected by trace amounts of surfactants. The packing size should be no larger than one-eighth of the tower diameter to prevent channeling. Packings of 1-1/2 in. (38 mm) size are common.

TOWER DIAMETER

Tower diameter for a packed liquid-liquid extractor will be set by one of four criteria: the two settling velocities for droplets at both ends of the extractor, coalescence time for the emulsion formed at the interface and the flooding limitation of the packing itself.

Droplet settling (or rising) rates may be calculated by the rigid sphere settling equations which apply to a single droplet settling in a stagnant liquid. These are the same equations used to size sieve tray extractors in [Section IV-B](#). Tower diameter is set such that the velocity of the phase from which the droplets are settling (or rising) does not exceed the droplet settling velocity, in order to allow the droplets to separate at the outlet zones of the tower.

Typically extractors are designed for essentially no entrainment and a design droplet size of 0.005 in. (0.13 mm) is chosen. For example, in amine treaters, entrained hydrocarbon in solvent could cause foaming in the amine regenerator, and entrained solvent in hydrocarbon product increases solvent makeup costs. Even when water washing facilities are provided after product treaters, it is generally desirable to minimize solvent losses to the water phase. For cases where some entrainment may be tolerable, such as extraction towers followed by entrainment removal facilities, a drop size larger than 0.005 in. (0.13 mm) may be used for calculating the limiting settling rate of that phase. It is recommended that the Fractionation and Thermodynamics Section of ER&E be consulted in such cases.

In addition, tower diameter must be adequate for coalescence area requirements, usually stated as a maximum dispersed phase volumetric flow rate per unit of tower cross-sectional area. A maximum of 10 gpm/ft² (6.8 dm³/s/m²) is used to avoid undesirably deep emulsion bands at the top of the tower. Poor separation and/or phase inversions can result from deep emulsion bands, and lead to flooding and entrainment. As discussed later in this section, coalescence media may be desirable to promote separation in some applications.

	EXTRACTION TOWERS PACKED EXTRACTION TOWERS		DESIGN PRACTICES	
	PROPRIETARY INFORMATION - For Authorized Company Use Only		Section IV-C	Page 5 of 20
			Date December, 1999	

DESIGN CONSIDERATIONS (Cont)

Finally, the tower diameter may be based on the flooding limitations of the packing itself. The variables which have typically been correlated with flooding of packing are physical properties, packing size, packing type, and phase flow rates. However, packing flooding is also a function of direction of mass transfer and which phase is dispersed. There is no simple correlation that accurately accounts for all these variables. The approach taken here is to use a simple correlation and reduce the predicted flooding velocities to account for known inaccuracies. The Crawford and Wilke (2) extractor packing flood curve shown in **Figure 2** should be used to estimate the maximum flow rate for a packing. Only 40% of this maximum rate should be used to account for uncertainties in the correlation. The Fractionation and Thermodynamics Section of ER&E can assist in determining interfacial tension data for various systems and provide consulting if low interfacial tensions, below 10 dyne/cm (10 mN/m), are anticipated.

TOWER HEIGHT

Height Equivalent of a Theoretical Stage (HETS)

No correlation has thus far been developed to predict accurately the Height Equivalent of a Theoretical Stage (HETS) for commercial packed extractors. In theory calculations similar to those discussed under **Section III-G** are possible, although the mass transfer coefficients would be different from those used for vapor-liquid systems. The University of Texas at Austin, as part of the Separation Research Program (SRP), has developed a computer program (7) to estimate packing efficiency; however as discussed below, it is likely to give optimistic estimates.

One problem in extraction is that mass transfer coefficients can be strongly affected by trace amounts of surfactants (9). Therefore, laboratory studies of mass transfer with chemically pure systems could overestimate commercial mass transfer.

Another problem is that most extraction data available for developing correlations have been obtained on small scale laboratory columns. Backmixing of the continuous phase becomes worse in packed towers as column diameter increases and obtaining more than one theoretical stage per packed bed is difficult. In addition, HETS is a function of packed bed height. Since the effect of column diameter and packed height have not been adequately researched, conservative design is recommended.

Most confidence is placed in HETS estimates obtained from existing commercial extractors in similar service. However, to provide guidance, the following table can be used for screening:

PALL RING SIZE		HETS	
in.	mm	ft	m
1.0	25	6	2
1.5	38	8	2.5
2.0	50	10	3

The number of theoretical stages (T_T) is calculated using the Kremser Equation (4) or by a stage-to-stage computer program such as Simulation Sciences' PRO II. The Fractionation and Thermodynamics Section should be consulted for the needed thermodynamics data, and for HETS data for commercial extractors.

Liquid Redistribution

Due to droplet coalescence and backmixing of the continuous phase, the dispersed phase must be redistributed to maintain good efficiency. The maximum packed bed height is 5 column diameters or 10 ft (3 m), whichever is less. Commercial packed bed heights are typically from 5 to 10 ft (1.5 to 3 m) tall, with 8 ft (2.4 m) being common.

Interface Detection and Control

Interface detection and control in extractors can be a problem, particularly when a wide emulsion band occurs at the interface. This emulsion build-up, and subsequent carryover, could be due to high dispersed phase flow rates or slow coalescence rates caused by contaminants such as surface active chemicals or fine particles. Interface measurements in gage glasses will indicate an average composition (density) of emulsion and liquid phases between interface taps, rather than an actual interface. In order to better establish the location of the coalesced layer the use of a series of overlapping gage glasses (minimum of 3) is recommended rather than a single gage over the entire interface control zone.

For control purposes, the height of the interface control zone, between the high (HIL) and low (LIL) interface levels in **Figure 1**, is typically set from the larger of 4 minutes hold-up based on the dispersed phase flow rate and 10 minutes hold-up based on the continuous phase flow rate. Consideration should be given for higher holdups in emulsion prone systems, or to take into account process or site-specific needs for dispersed or continuous phase holdups. For amine systems, there should be at least 3 ft (1 m) between the high and low liquid-liquid interface level (11).

DESIGN PRACTICES		<i>EXTRACTION TOWERS</i> PACKED EXTRACTION TOWERS PROPRIETARY INFORMATION - For Authorized Company Use Only	
Section	Page		
IV-C	6 of 20		
Date December, 1999			

DESIGN CONSIDERATIONS (Cont)

Capacity for at least 2 minutes of holdup of dispersed phase (4 minutes for amine systems) is recommended between the high interface level and the top tangent of the vessel. This space provides extra holdup for operation response in case the interface is lost above the high interface level. The minimum height of this space should be 2 ft (0.6 m) to allow for a future installation of coalescence media.

The interface level is typically maintained by controlling the heavy phase outlet flow. As shown in **Figure 1**, the feed hydrocarbon (light phase) and solvent (heavy phase) are on flow rate control with the solvent rate being some fraction of the hydrocarbon rate.

TOWER INTERNALS

Packing Supports (Dispersers and Redistributors)

Modern packing supports for extractors also typically serve as dispersers or redistributors. An example is shown in **Figure 3** for use when the hydrocarbon phase is dispersed. Vendors usually design the size, number, and layout of the small holes for hydrocarbon flow as well as the size, number, and layout of the downcomers for the continuous aqueous phase. Vendor designs should be checked against the following guidelines (assuming hydrocarbon phase is the dispersed phase). Deviations from these guidelines should be checked by the Fractionation and Thermodynamics Section of ER&E.

- Hole diameter for hydrocarbon flow should be 0.25 in. (6 mm).
- Depth of hydrocarbon layer should not exceed downcomer length at maximum rate. 12 in. (300 mm) is the typically preferred downcomer length to provide good distribution and volume for coalescing emulsion. For checking designs, depth of hydrocarbon layer can be calculated from:

$$b = \frac{\rho_D}{2 g \Delta \rho} \left(\frac{V_D}{60 A_H C} \right)^2$$

where: A_H = Total hole area for dispersed phase flow, ft² (m²)

b = Depth of dispersed phase layer below holes, ft (m)

C = Coefficient of discharge = 0.5 (Note this is an estimated coefficient adjusted to approximately account for the effect of packing on top of the holes, downcomer velocity and interfacial tension effects.)

g = Gravitational constant = 32 ft/s² (9.8 m/s²)

ρ_D = Density of dispersed phase at operating conditions, lb/ft³ (kg/m³)

$\Delta \rho$ = Density differential between the dispersed and continuous phases (absolute value), lb/ft³ (kg/m³)

V_D = Volumetric flow rate of dispersed phase, ft³/min, (m³/min)

- Hydrocarbon hole velocity should be less than 2 ft/s (600 mm/s).
- Continuous phase downcomer velocity should be less than 0.5 ft/s (0.15 m/s).
- There should be at least 0.7 downcomers per ft² (7 downcomers per square meter).

If the heavy phase were dispersed, the disperser/redistributors would look similar to that shown in **Figure 3**, but would be turned upside down. In addition, the support for the bottom packed bed would be similar to the sinusoidal-shaped gas injection support plate used in distillation.

Bed Limiters

A bed limiter (packing hold down screen) should be specified for each bed to contain the packing during an upset. They should be fastened to the vessel with wall clips. For breakable (ceramic) packing, a non-supported type of bed limiter should be specified for each bed to prevent packing movement.

Inlet Distributors

Vendors usually design the distributors; however, the design should be checked to ensure that hole velocities are less than 3 ft/s (900 mm/s), preferably less than 1 ft/s (300 mm/s), and hole sizes are at least 1/4 in. (6 mm) in diameter.